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TANTALUM ALLOY TUBING DEVELOPMENT PROGRAM

Allegheny Ludlum Steel Corporation
Research Center

Contract AF 33(657)-11261
RTD Project: 8-109 (I)

First Interim Technical Engineering Report

1 July 1963 to 30 September 1963

A state-of-the-art analysis has been completed covering potential application for, domestic and foreign technology used in the production of, and a review of mechanical and physical property data on tantalum alloy tubing. Usage was made of a questionnaire, personal visitations and/or contacts and a review of domestic and foreign literature. The survey revealed that the primary potential utilization of tantalum alloy tubing is in nuclear aerospace propulsion and auxiliary power systems. The proposed program for Phases II, III and IV is submitted.

METALLURGICAL PROCESSING BRANCH
MANUFACTURING TECHNOLOGY DIVISION
AIR FORCE MATERIALS LABORATORY

Research and Technology Division
Air Force Systems Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

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FOREWORD

This First Interim Technical Engineering Report covers work performed under Contract AF33(657)-11261 from 1 July 1963 to 30 September 1963. It is published for technical information only and does not necessarily represent the recommendations, conclusions or approval of the Air Force.

This contract with the Research Center of the Allegheny Ludlum Steel Corporation, Brackenridge, Pennsylvania, was initiated under RTD Manufacturing Technology Division Project 8-109, "Tantalum Alloy Tubing Development Program." It is administered under the direction of Mr. H. L. Black of the Metallurgical Processing Branch (MATB), Manufacturing Technology Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio.

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ABSTRACT - SUMMARY
First Interim Technical Engineering
Report

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A State-of-the-Art analysis has been completed covering potential application for, domestic and foreign technology used in the production of, and a review of mechanical and physical property data on tantalum alloy tubing. Usage was made of a questionnaire, personal visitations and/or contacts and a review of domestic and foreign literature. The survey revealed that the primary potential utilization of tantalum alloy tubing is in nuclear aerospace propulsion and auxiliary power systems. The proposed program for Phases II, III and IV is submitted.

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TANTALUM ALLOY TUBING DEVELOPMENT PROGRAM

State-of-the-Art-Analysis

INTRODUCTION

One of the major components of advance nuclear aerospace units is tubing of a satisfactory quality to meet power generation requirements involving liquid metals. Items such as boilers, radiators, and turbine nozzles for use in both nuclear propulsion and nuclear auxiliary power units will consume relatively large quantities of small diameter thin wall tubing.

The refractory metals find application in these boiling-metal systems at temperatures appreciably lower than what is considered their normal use temperature in conventional high-temperature structural applications by virtue of their unique corrosion resistance to molten alkali metals. However, the size and weight of the radiator varies inversely as the fourth power of the radiator temperature, so that small gains in temperature produce large weight reductions. Consequently, considerable effort will be directed toward raising operating temperature to achieve both weight reduction and higher power output and thus making the use of refractory metal alloys mandatory.

Recognizing the potential shown by tantalum alloys for this application, the Aeronautical Systems Division of the United States Air Force awarded to Allegheny Ludlum Steel Corporation Contract AF33(657)-11261, a "Tantalum Alloy Tubing Development Program." The specific objective of the program is to provide processing mechanisms and sequences for the production of defect-free, fabricable and weldable tantalum alloy tubing displaying improved high strength and corrosion resistance characteristics. The program involves four chronological phases:

- Phase I - State-of-the-Art Analysis
- Phase II - Development of Tube Hollows
- Phase III - Development of Tube Production Process
- Phase IV - Production of Tubing

This report constitutes the effort under Phase I. Since items such as raw materials, ingot consolidation, initial ingot breakdown, etc., were thoroughly covered in State-of-the-Art Analysis conducted for previous contracts involving tantalum alloys [1] [6], it was requested by the Air Force personnel that special emphasis be placed on applications for tantalum alloy tubing and the metallurgical problems and difficulties unique to these applications. However, since the optimum factors in tube production are closely inter-related and a selection of one cannot be made without careful consideration of its influence on all the other factors, the survey was made as comprehensive as possible.

In conducting the survey use was made of a questionnaire, personal visits or contacts and an extensive search of both the domestic and foreign literature.

The questionnaire, which is reproduced in Appendix A to this report, was mailed to 108 industrial concerns, research organizations and Government agencies known or believed to have applications for or experience in the production of tantalum

alloy tubing. The list of organizations contacted is given in Appendix B. Of the 108 questionnaires mailed a total of 60 replies were received. Only 22 of these contained useful information.

The American Society for Metals, Information Searching Service, was contracted to perform a retrospective complete file search for the years 1958 to date. A total of 67 abstracts were obtained from this search. A manual search was made of the period prior to 1958 but no significant references were located.

The foreign technology phase of the program was conducted by World Progress in Engineering, Inc. A report of their findings is given in Section II of this report.

SUMMARY AND CONCLUSIONS

Results of this survey indicate that the greatest potential for tantalum alloy tubing is in the nuclear aerospace applications, especially in nuclear auxiliary power generation systems involving liquid metals. Large quantities of small diameter thin wall tubing are required in the boilers and radiators of these systems.

The properties most important to these applications are (1) corrosion resistance to liquid alkali metals at temperatures up to 2400F, and (2) weldability and metallurgical stability over long periods of time at temperatures up to 2400F.

At least three producers list tantalum alloy tubing as items in their commercial product line. However, only relatively small quantities have actually been produced, and there is little test data available on that which has been produced. The potential users feel that the tubing currently produced is not of a consistently high quality and report wide variances in the quality from batch to batch. Consequently, every step in the production of tubing must be closely controlled to assure a uniform reproducible high quality.

In the United States powder metallurgy methods have virtually ceased except for the fabrication of electrodes for subsequent melting. In Europe the powder metallurgy techniques of producing tantalum and tantalum alloy products are still of prime importance.

Most experience in melting ingots of three-inch diameter or larger is with the Ta-10W and T-111 (Ta-8W-2Hf) alloys. Ta-30Cb-7.5V is being used on a sheet rolling program but only a limited number of billets have been made. Double melting is the general practice whether it be by electron beam or consumable arc or a combination of the two. Many European firms have recently or are currently setting up for electron beam melting. Consumable arc melting of tantalum alloys is not practiced in Europe.

Initial conversion of the Ta-10W and T-111 and Ta-30Cb-7.5V alloys can be accomplished by either forging or extrusion. Forging practices are quite uniform throughout the industry. Forging temperatures are in the range of 1800-2200F. Billets of Ta-10W as large as 8-1/2-inch diameter have been extruded. Reduction ratios for initial breakdown are usually about 4:1. Temperatures for breakdown extrusion varied between 2000 and 3100F. Billets are either clad in another metal or coated with a glass slurry prior to heating to eliminate surface oxidation or heated bare in an inert atmosphere.

The main difficulty in the production of tantalum alloy tubing is in obtaining high quality tube hollows. Of the many methods of obtaining tube hollows, direct extrusion offers the most economical and practical means. For this reason our program will concentrate on the development of the extrusion method of producing high quality tube hollows.

Recommended stress relief anneal for all alloys is one hour at 2000F. For 100 percent recrystallization one hour at 3000F is generally used, although the actual recrystallization temperature is usually lower than this and can be achieved at lower temperatures for longer times. All heat treating must be done in vacuum.

Most of the mechanical property data available is on the Ta-10W and T-111 alloys. Limited data is available on Ta-30Cb-7.5V. Preliminary data on T-222 (Ta-9.6W-2.4Hf-.01C) i- :cated that this alloy has higher strengths than either the Ta-10W

or T-111 alloy at elevated temperatures and is as easily worked.

Ta-10W, Ta-30Cb-7.5V, STA-900, T-111 and T-222 can be easily fussion welded and welded areas display excellent room temperature bend ductility with no significant loss in room temperature tensile properties. The T-111 was completely ductile to temperature of -320F while the preliminary data on T-222 show the alloy to be completely ductile at -250F but brittle at -320F.

SECTION I

Interests, Applications, and Requirements for Tantalum Alloy Tubing

INTRODUCTION

Section I of the questionnaire was concerned with interests, applications and requirements for tantalum alloy tubing. The replies to this portion of the questionnaire are summarized in Table I. Of the 60 replies received only eight organizations reported current needs for tantalum alloy tubing, although it is known that many organizations are using small amounts for items such as heating susceptors and heat exchanger tubing. Besides these common items most of the present requirements are experimental in nature and used in various tests assimilating future applications.

Most of the proposed applications for tantalum alloy tubing are involved in advanced nuclear aerospace propulsion and power generation systems. A brief review of these experimental or proposed systems and of the components which may utilize tantalum alloy tubing is given in the following paragraph:

Nuclear Aerospace Propulsion - Project Rover

Project Rover is the code name of a coordinated AEC-NASA program for the development of qualified nuclear rocket engines for the accomplishment of operational space missions. Under this program the AEC is responsible for the development of reactors and reactor technology and the NASA is responsible for the development of the nuclear engines and related engine technology, and for the integration of the reactors into engines.

In propulsion devices thrust is generally realized by acceleration of some kind of matter through a nozzle. A chemical rocket utilizing a fuel and an oxident achieves high-velocity exhaust mass by combustion of the propellants causing high temperatures with resultant expansion and high velocity of the combusted gases and solids. Thermal rockets which offer higher specific impulses (i.e., pounds of thrust per pound of propellant per second) use a single propellant that is heated by some auxiliary heating method, either a nuclear reactor or electrically heated elements. Hydrogen in the liquid state is the most attractive propellant for these thermal rockets, since the exhaust velocity and associated thrust increases as the molecular weight of the propellant decreases. It is reported[2] that with hydrogen at a temperature of 4700F a specific impulse of 1000 pounds per pound per second is possible. Finger[3] states that the nozzle in a thermal rocket engine will experience heat fluxes 50-100 percent higher than the heat fluxes in chemical rocket engines. In view of this the nozzles will be a major problem area in advanced nuclear rocket engines.

Advanced research and technology on engine systems components, such as advanced flow systems and nozzles, is centered at NASA's Lewis Research Center, with supporting effort by various industrial contractors. Since liquid hydrogen will be available in the system NASA proposes to utilize this to regeneratively cool

TABLE I

Interests, Applications and Requirements for Tantalum Alloy Tubing

Needs for Tantalum Alloy Tubing	Number of Affirmative Replies
At Present	8
In the Near Future	12
In the Distant Future (five years or more)	18

Potential Applications

Liquid Metal Cooling Systems
Nuclear Rocket Engine Thrust Chamber
Regenerative Cooled Chemical Rocket Engine
Fluid Transfer in Non-Structural Systems
Thermionic Diodes
Heat Exchanger Tube
Thermocouple Sheathing
Molten Plutonium Fuel Capsules
Heating Susceptors

Properties or Characteristics Required

Resistance to Liquid Metal Corrosion
Compatibility with Rocket Engine Exhaust Products
Weldability
Bendability
Metallurgical Stability Over Period of Years at Temperatures of 2000-2200F
Tensile and Creep Properties Equivalent to or Better than those for the Cb-1Zr Alloy

the nozzle. The two nuclear propulsion reactors currently under development, KIWI and NERVA, use Inconel X and stainless steel tubing, respectively, for this purpose. In advance nuclear rockets of the future the refractory metals would have to be considered for this application because of the higher heat fluxes anticipated. Consequently, tantalum alloys and columbium alloys are currently being studied for use. A sketch of this nozzle design is shown in Figure 1. The nozzle would consist of tantalum or other refractory metal alloy tube welded together in the shape of a cone with an outer skirt of nickel or some superalloy. The liquid hydrogen would be pumped into the tube at a rate of approximately 800 pounds per second, absorb heat from the exhaust, boil and enter the reactor as a gas. The temperature in the reactor would be about 4500-5000F. The temperature inside the tubes will be 3000 to 3500F with a gas pressure of approximately 1000 psi. The size contemplated for this application is about 1/4 inch square by .020 inch or less wall thickness. The required life will be a minimum of three firing periods of about 1/2 hour each.

In addition to the needs for power for propulsion, electric power is required in space for the operation of communications systems, scientific equipment, etc. Our space vehicles currently get their power from solar or chemical batteries which are neither light weight nor inexpensive. Obviously, because of the great cost and difficulty of launch, a basic parameter for evaluation and comparison for any component to be used in space is its weight. Figure 2 illustrates the specific weight of various power generation systems in pounds per kilowatt as a function of electrical power output in kilowatts. This figure clearly shows that if we are to achieve maximum power output at minimum specific weight for future spacecraft emphasis should be on the reactor turbo-generator system. Figure 3 is a schematic sketch of such a system.

Under the joint efforts of AEC and NASA the SNAP 2 and SNAP 8 systems are currently under development, and will produce 3 and 30 kw of power, respectively. A joint AEC-NASA-DOD project is currently under way to develop and provide advanced technology for a power plant operating in space environment and having electrical power output of 1 megawatt.

Two reactor turbine generator systems under consideration for the more advanced power plants are the Rankine cycle and the Brayton cycle. The Rankine cycle is a standard turbine generator system in which heat is generated in a compact nuclear reactor. In this system the water normally used as the cycle working fluid is replaced by a more efficient liquid alkali metal. The liquid metal is heated by the reactor, is converted into a vapor which is used to drive the turbine which in turn drives a generator to produce the electrical power required. The vapor is then condensed to a liquid and pumped back to the reactor. Since, in space,cycle waste heat must be rejected by radiation, a radiator must be used in conjunction with the condenser. The metals under consideration for use as the cycle fluid are sodium, potassium, rubidium, cesium and lithium. Different fluids are of interest for different temperature levels by consideration of their vapor pressures. Naturally, the most important consideration in selecting metals for the components of this system is their compatibility with the liquid metals used. Maximum temperatures will vary between 1800-2400F depending on the cycle fluid used. It is reported[2] that presently available data indicates that the superalloys would be severely corroded by the working fluids at these temperatures. Consequently, the refractory metals, by virtue of their unusual corrosion resistance to molten alkali metals, will be required for this application. Stress will be in the neighborhood of 6000 psi.

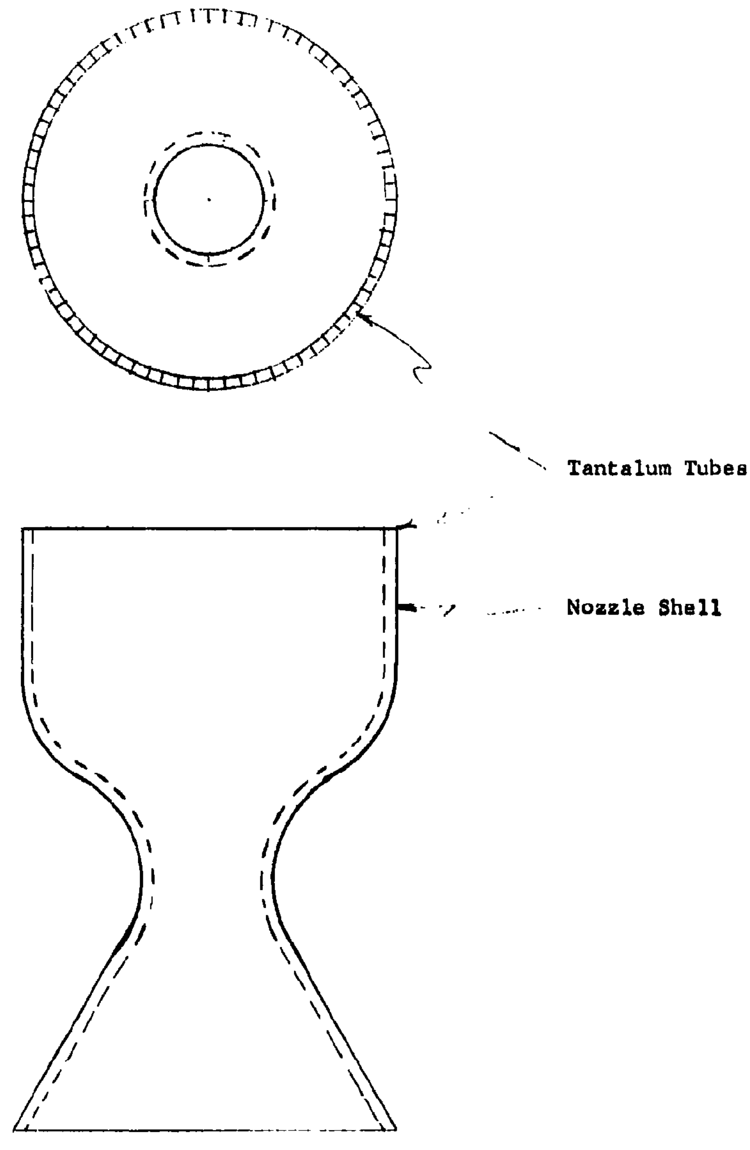


Figure 1

A proposed nozzle design for advanced nuclear rockets. Liquid hydrogen flowing through tantalum tubing used to regeneratively cool nozzle shell.

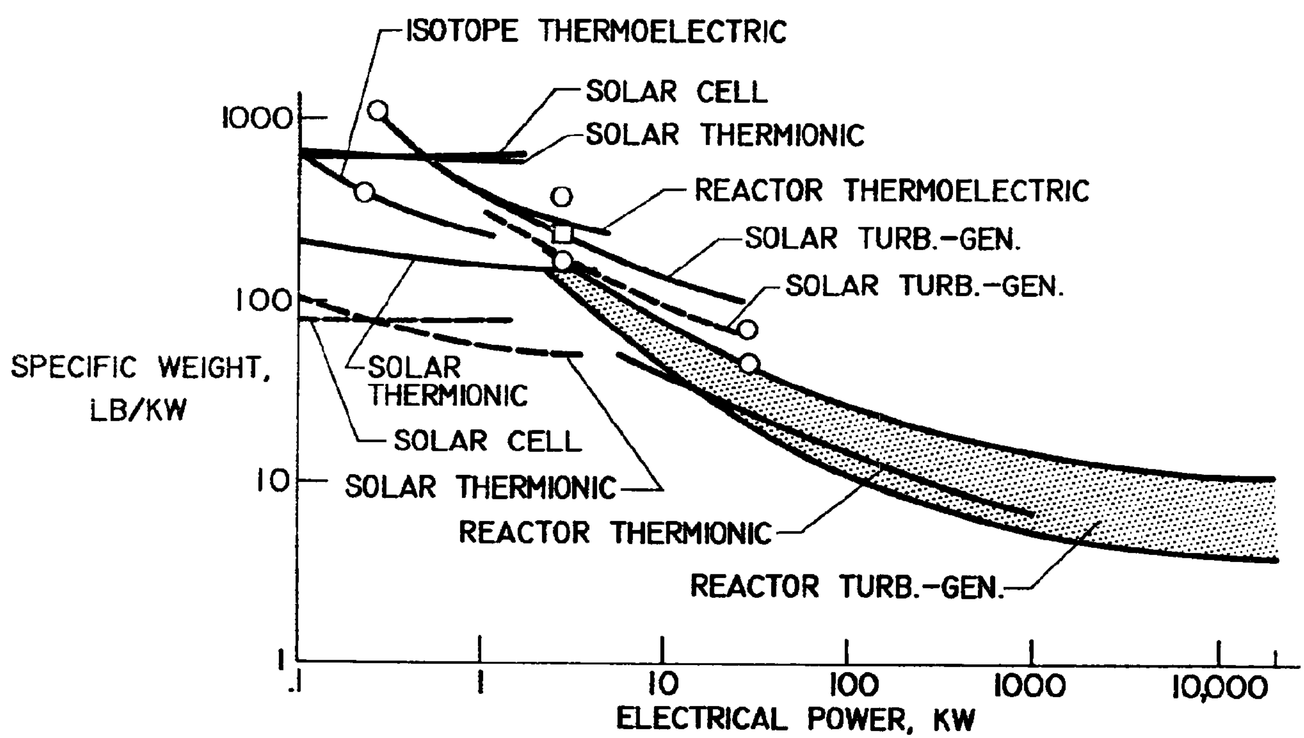


Figure 2

Estimated specific weights of power generator systems

(Courtesy of NASA)

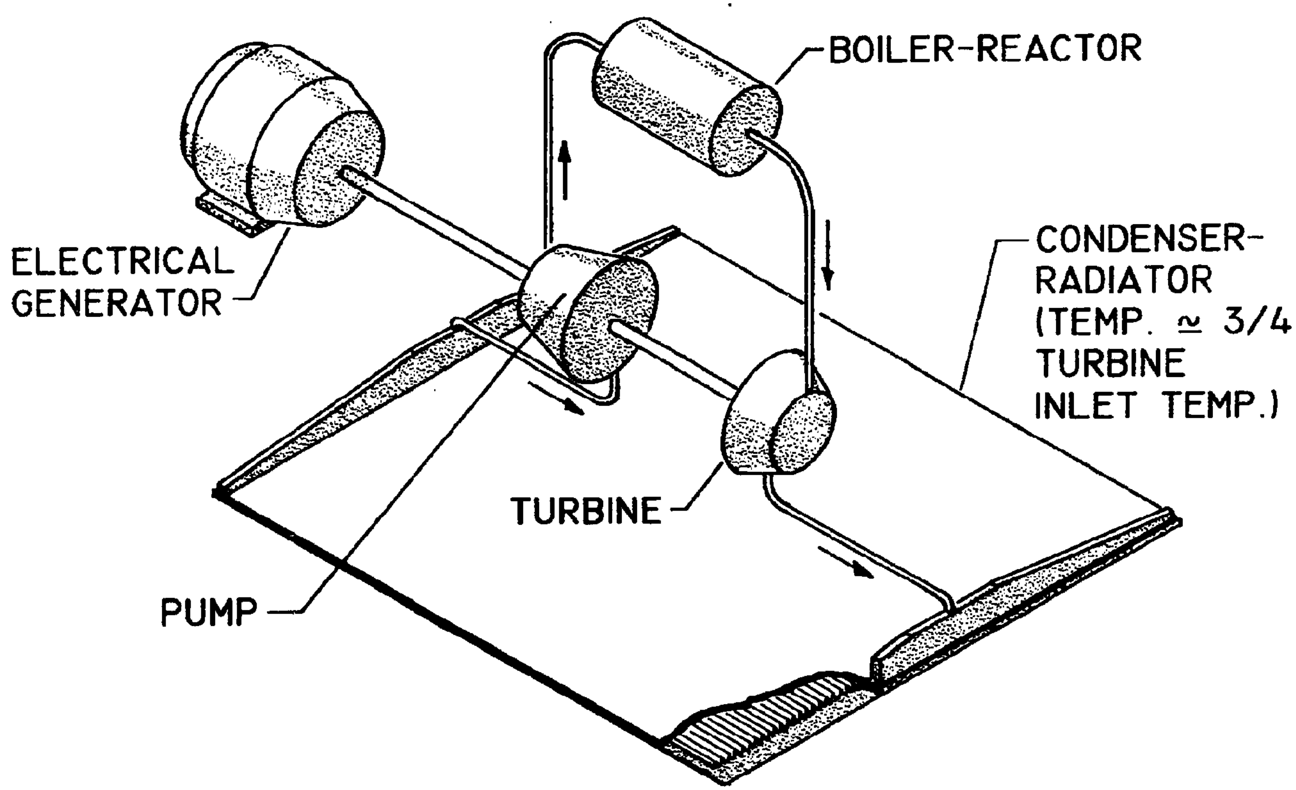


Figure 3

Nuclear reactor turbo electric system (Single Loop)

(Courtesy of NASA)

The Brayton cycle uses a neutral gas such as helium, argon or neon rather than a liquid alkali metal as the cycle fluid. In this system, the temperature of the gas going into the turbine is about 2500F. The peak temperatures in the radiator will be about 2000-2200F. While the Brayton cycle eliminates the corrosion problem, the size of the radiator required is about ten times that required for the Rankine cycle.

In reactor turbine generator systems the radiator alone accounts for 40-60 percent of the total weight. Figure 4, a schematic drawing of a manned vehicle of this general type illustrates this point. Large quantities of tubing are required for use in the boiler and radiator. Ault(2) estimates that for a 1000 kw system the boiler, if made of 1/4 inch diameter tubing, would require 3300 feet of tubing and the radiator, if likewise made of 1/4 inch diameter tubing, would require about 55,000 feet. As previously stated in the introduction, the size of the radiator varies inversely as the fourth power of the radiator temperature. Consequently, if greater powers are to be realized from these systems, operating temperatures must be raised to reduce specific weights. It is at the higher temperatures that tantalum alloys would have the advantage over all other materials under consideration. In addition to the large amount of tubing required in the boiler and radiator, relatively small amounts of interconnecting pipe and tubing in the size range of 2-inch to 20-inch O.D. are required.

Another application listed by the Jet Propulsion Laboratory of California Institute of Technology is a thermionic diode material in a thermionic converter system. In this system a cathode is heated to temperatures at which it will emit electrons which pass through a vacuum or cesium vapor and will collect on a cooler anode. An advantage of this system is the possibility of radiating waste heat directly from the anode. However, for comparable power output the thermionic systems must operate at higher temperature than the turbine-generator systems. This may make radiation directly from the anode impossible and it may be necessary to employ a liquid metal cooling loop to extract heat from the anode and carry it to a radiator. If this liquid metal cooling system is required then large amounts of tantalum alloy tubing could be required.

Tantalum tubing is currently being used as "thimbles" or capsules for the molten plutonium fuel in the LAMPRE-I fast reactor experimental core facility at Los Alamos Scientific Laboratory. This reactor is part of a program to determine under what conditions and forms plutonium can be used as fuels in power reactors. The capsules are 0.375 inch I.D. by .020 to .030 inch wall by 9 inches long and closed at one end. The capsules for this reactor are currently fabricated at the laboratory because of lack of interest by commercial fabricators.

Tantalum was chosen for this application because it was one of the few metals available capable of containing molten plutonium for an appreciable period of time and was the most fabricable of those considered. The existing one megawatt reactor uses about 250-300 feet of tubing per year. A fifteen megawatt core test facility reactor which would require 760 capsules 16-18 inches long is in the planning stages. For this reactor 1500 to 2000 feet per year would be required plus about 1000 feet per year for developmental testing purposes.

The temperature in the reactor during run is about 1120F. Plutonium freezes between 660 and 750F and undergoes about 1-3 percent expansion on freezing. This causes distortion in the unalloyed tantalum capsules presently in use. Experimental data indicates that the Ta-10W alloy is strong enough to withstand this distortion. Strength was not of prime consideration in selecting the material,

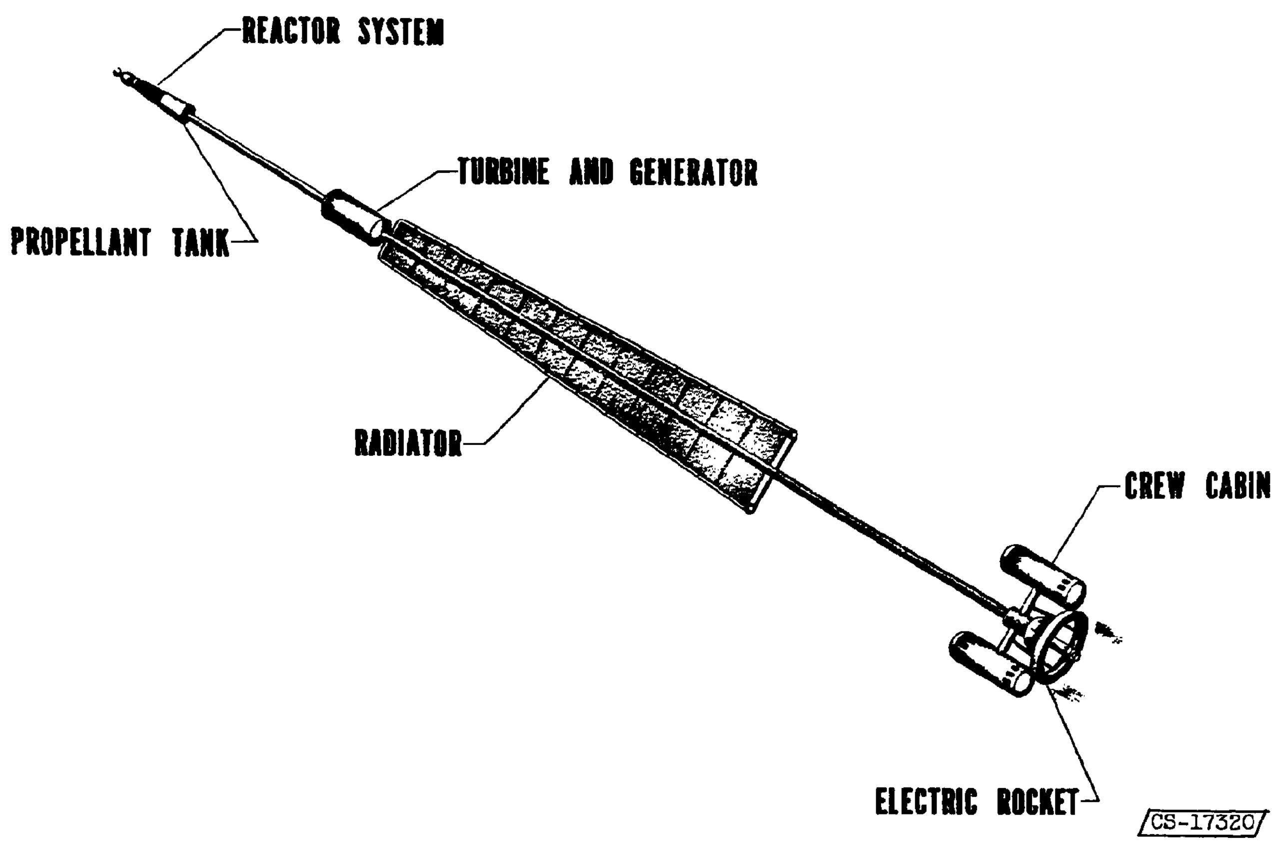


Figure 4

Electric Space Vehicle

(Courtesy of NASA)

since they are able to design the reactor to suit the materials used. However, provided the alloying elements do not deter corrosion resistance, the higher strengths made possible by alloying will make recycling of the reactor possible.

Relatively small quantities of tantalum tubing in a large variety of sizes are used in the chemical industry for fluid transfer, since tantalum and its alloys are extremely resistant to all acids except HF and acids containing free sulfur trioxide. It is even resistant to these except in high concentrations and at high temperatures.

Vogel[4] reports that tantalum and its alloys are probably the most efficient heat transfer materials used in the process industries for corrosive environments. While tantalum tubing is used in conventional shell and tube type heat exchangers, its most promising application in this field is in the form of a bayonet heater. Here it is usually less expensive for an equivalent heat transfer surface.

Obviously, from the nature of the applications mentioned above, the most important property of the tantalum alloy tubing is corrosion resistance to its service environment, whether it be liquid alkali metals, liquid plutonium, acid salts, or rocket engine exhaust products. Other properties sited in the replies to the questionnaire are bendability, weldability, and metallurgical stability over a period of years at temperatures between 2000-2200F. Since it is believed that tubing will fail by bulging or creeping longitudinally rather than fracturing 10,000 hour creep data is required.

While tantalum alloy tubing is currently available in experimental quantities, insufficient information is available on its properties and behavior to adequately judge its quality. Los Alamos Scientific Laboratory reports wide differences in the quality of commercial tubing from batch to batch and from fabricator to fabricator. They have received tubing of excellent quality from one vendor and the next shipment from the same source was inferior, in that it contained laps and seams emanating from the O.D.,and small trapped impurities were found on the O.D. and I.D. surfaces of the tubing. Figure 5 is a photograph showing the I.D. and O.D. surfaces of tubing purchased from a commercial vendor. It is obvious from this that every step in the fabrication from starting material to finished tube must be closely controlled to obtain uniform reproducible high quality tubing.

RAW MATERIALS

The most important source of tantalum metal is the mineral tantalite (also known as columbite). The ores are treated chemically to produce pure compounds which can be reduced to metal. The most common method employed is a liquid-liquid extraction to form an oxide which is reduced to metal either by carbon or sodium reduction. A more detailed summary of the available ores and the various methods of extracting the metal from the ores is given by Wah Chang Corporation in an earlier state-of-the-art survey on Contract AF33(600)-42396[6].

Metal powder is the most important primary form of tantalum for use in tantalum base alloys. Tantalum powders are commercially available in a variety of grades and sizes with the average particle size about -60 mesh. The metallic impurity level of the as-reduced powder is very low. The impurity levels for carbon, oxygen, hydrogen and nitrogen are dependent on the reduction process and can vary considerably. The maximum impurity levels in tantalum powders used for melting, as reported by the producers, are given in Table II. It was generally felt that the interstitial levels in the alloying elements used were not too important,

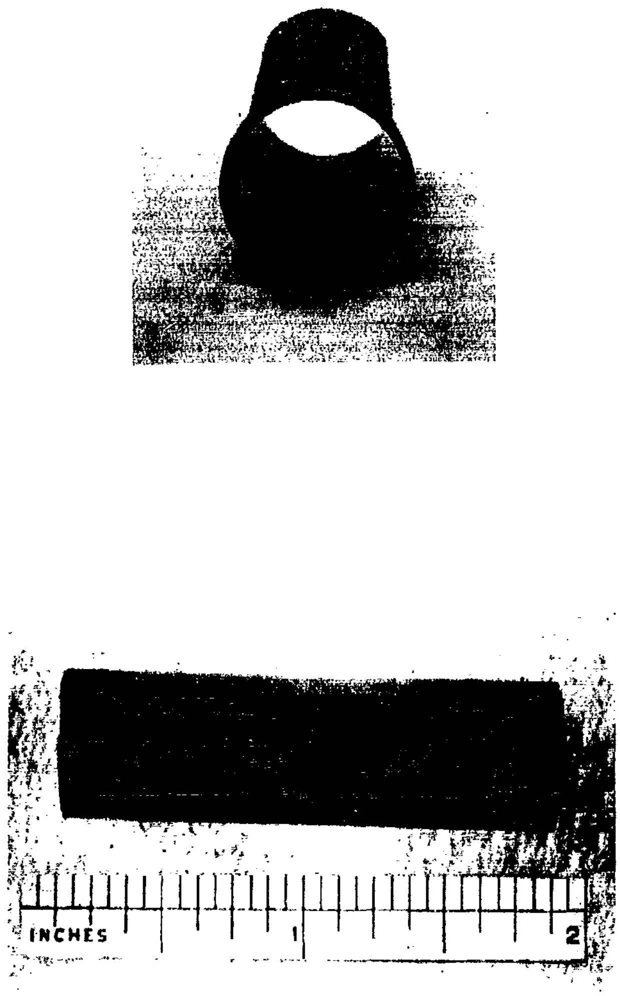


Figure 5

Photographs of tubing purchased from a commercial vendor showing trapped impurities

TABLE II

Maximum Impurity Levels for
Tantalum Powder of Melting Grade

Impurity	Maximum Impurity Content % Fansteel Metallurgical Corporation	Kawecki Chemical Company	National Research Corporation	Herman C. Starck* Goslar, Germany	Wah Chang Corporation
C	.050	.02	.0075	0.1	.010
N		.01	.0050	0.03	.020
O		.18	.0450	0.2	.150
H			.0075	0.05	.005
Cb	.070	.07	.0075	0.5	.010
Fe	.020	.02	.0045	0.1	.010
Ni		.01	.0100	0.01	.002
Si	.010	.015	.0250	0.03	.025
Ti	.010	.010	.0040	0.05	.002
W		.02	.0040	0.01	.002
Others			.0050	0.05	.002

*Information received through Shieldalloy Corporation.

since the impurities can be removed by solid state purification and consumable arc or electron beam melting in high vacuum.

The price of tantalum powders is dependent upon the specifications and delivery required, but currently falls in the range of \$28.00 to \$42.00 per pound.

CONSOLIDATION OF RAW MATERIALS

In the early stages of development fabrication of products from tantalum alloys was accomplished using powder metallurgical practices. However, with the successful adaptation of the consumable electrode and electron beam melting techniques to tantalum alloys, direct fabrication of compacted tantalum alloy billets has been discontinued in the United States. None of the producers contacted supply pressed and sintered billets for direct forging or extrusion.

Virgin charges for both the consumable electrode arc and electron beam melting procedures are normally prepared from powders and the electrodes for both processes are formed by powder metallurgy techniques.

The initial step in the preparation of electrodes is the blending of the tantalum and alloy powders. This is accomplished either by hand or in any of the numerous blending devices on the market. While most of the companies consider the compacting details proprietary, NRC reports that the Ta-10W alloy is compacted in either a mechanical or hydrostatic press using 30,000 psi pressure. Wah Chang's procedures for this alloy are detailed in the second interim report on Contract AF33(600)-42396[7]. Both companies report the as-pressed densities to be about 70 percent.

Following pressing, the green bars are generally, though not always, sintered. Sintering is usually done in vacuum by direct resistance heating. The sintering procedures are likewise considered proprietary by most concerns. NRC reports the sintering cycle as ten minutes at 1600C. They sinter only for the purpose of strengthening and improving conductivity. Purification can be accomplished during vacuum-direct-resistance sintering. Klopp, et al[8], have reported the reactions occurring during sintering of tantalum. Essentially, the tantalum carbide TaC reaction with tantalum oxide Ta_2O_5 in vacuum begins at 1400C and evolution of CO continues to at least 2200C. Initial ratio of oxygen to carbon has a significant effect on the final composition of the sintered bars. Based on these findings, Wah Chang, in the previously referenced report[7] selected the following sintering cycle: Steady heating at a vacuum of .2 microns for three hours to reach 1700C then increase temperature in 100C increments to 2400C. Approximately one-half hour was required for each 100C increment and temperature was held for one hour before raising controls to next increment. Table III illustrates the effectiveness of this sintering cycle as reported by Wah Chang[7].

MELTING TECHNIQUES

All tantalum alloys are melted in vacuum by either the consumable electrode arc melting or the electron beam melting process or a combination of the two. Only two tantalum alloys, the Ta-10W and the Ta-8W-2Hf, can be considered to be commercially available. These two alloys are available from a number of sources in a variety of ingot sizes. The Ta-30Cb-7.5V alloy is being used on some government-sponsored contracts but only Wah Chang Corporation seems to have had any experience

TABLE III*

Chemical Analyses of Starting Powders (Carbon Reduced) and Sintered Bar

Element	(Analysis in ppm) Ta Powder	Ta + 10%W Powder	Bar Sintered @ 2400C
O	2900	2700	660
C	152	140	< 30
N	350	340	75
Fe	580	580	155
Al	< 20	< 20	20
Si	655	600	125
Ti	< 20	< 20	< 150
Cb	< 500	< 500	
Zr			< 150
Hf			< 80
B	< 1	< 1	< 1
Cd	< 5	< 5	< 5
Cr	150	150	20
Cu	< 40	< 40	< 40
Mg	20	20	< 20
Mn	< 20	< 20	< 20
Mo	< 20	< 20	20
Ni	20	20	30
Pb	< 20	< 20	< 20
Sn	< 20	< 20	< 20
V	< 20	< 20	< 20
Zn	< 20	< 20	< 20

*Courtesy of Wah Chang Corporation

in melting this alloy, and trouble has been encountered in controlling the vanadium content. Table IV shows the various companies currently supplying tantalum alloy ingots, the alloys they produce and the maximum sizes available.

Both the consumable electrode and electron beam process utilize vacuum atmospheres and cold molds, both of which prevent undesirable reactions of the metals. Water cooled copper crucibles are generally used. Moderate vacuums are used in consumable electrode melting, but very high vacuums in the range of 0.1 to .01 microns are inherently required by the electron beam process. The heat for melting in this process is generated by bombarding the electrode or feed material and molten puddle with an intense beam of electrons and the presence of a plasma will short out the high voltage causing interruption of power.

While substantial purification has been obtained with consumable electrode vacuum arc melting, very extensive refining resulting in very low level of interstitials and volatile metallic elements are obtained with electron beam melting. This is possible by virtue of both the much higher vacuum and the fact that the beam can be controlled to keep a given volume of metal molten for an indefinite period. A comparison of the purification capabilities of the two processes is shown in Table V. This is a compilation of data previously reported[1] [9].

Consumable Electrode Melting

The electrode sections are generally TIG welded into either round or octagonal configurations, although rectangular shapes have also been utilized. The sections have also, on occasion, been joined by threaded nipples. The area ratio of electrode to crucible varies between 1:2 and 1:4.

While good homogeneous ingots have been obtained from a single melt, it is a general practice to double melt all tantalum alloys. The first melt is performed at a slow rate to obtain maximum purification, and the second melt is performed at a fast rate to obtain better homogeneity and improved ingot sidewalls and finer grain size.

While straight polarity, DC melting is the normal procedure employed by most melters, Ammon of Westinghouse reports[11] that they preferred AC power for primary melting the T-111 alloy because of the more uniform melt-off of material from the electrode. Stirring coils are normally employed to control arc stability. To avoid piping, hot topping, a procedure whereby the power is dropped back to some intermediate range and very slowly cut to zero power, is most generally used. Grain refiners and deoxidizing agents are generally not used. Stauffer did offer STA-900, a Ta-10W alloy containing a proprietary grain refiner, and NRC has a patent whereby carbon is used as a deoxidizing agent.

Billets as large as 10-inch diameter of Ta-10W alloy have been produced. One of the principle advantages of the consumable electrode vacuum arc melting process is its capability of almost unlimited scale-up to large size ingots. The reported normal yield from electrode to conditioned ingot is about 85 percent.

Electron Beam Melting

The starting material for EB melting can be in a variety of forms from loose powders, chips or turnings to compacted bars or massive scrap. This method is

TABLE IV

Availability of Tantalum Alloy Ingots

Company	Alloy Composition	Maximum Ingot Diameter(in.)	Maximum Weight (lbs)
Stauffer Metals	Ta-10W	8	1500
	T-111	8	1500
National Research Corporation	Ta-10W	6-1/2	400
	T-111	6-1/2	400
Wah Chang Corporation	Ta-10W	10	900
	Ta-30Cb-7.5V	9-1/2	850
Herman C. Starck Goslar, Germany	Ta-10W	2-3	
Fansteel	Ta-10W		

TABLE V

Comparison of the Purification Capabilities of Consumable Vacuum Arc Melting and Electron Beam Melting

	NATIONAL RESEARCH CORPORATION				WAH CHANG CORPORATION			
	Arc Melted		EB Melted		Arc Melted		EB Melted	
Impurity	Before Melting	After Melting	Before Melting	After Melting	Before Melting	After Melting	Before Melting	After Melting
C	100	25		12	100	30	100	<30
N	30	30		23	100	25	100	15
O	250	30	200-500	10	600	200	600	<50
H	50	Nil						1-2
Fe	30	10		<10				
Cr	20	<10		<10				
Ni	30	<10		<10				

All values given in ppm.

particularly adapted to the melting of scrap material. All melters, with the exception of Shieldalloy, use scrap in varying degrees, depending on the availability and the alloy to be melted. A slow melting rate is used to obtain optimum purification. The ingot thus obtained has a high purity level with poor sidewalls and a large grain size. This ingot can then be remelted by either the EB or consumable electrode process at a faster rate to improve sidewall condition and to obtain a finer grain size.

The melting rates possible with the EB process are considerably slower than those possible with consumable electrode arc melting and the grain sizes obtainable with EB melting are correspondingly larger. It is generally agreed that a fine grained cast structure is desirable for better workability. Consequently, it is often advisable to use a duplex melting system, the primary melt being EB and the secondary consumable electrode vacuum arc. By this process electron beam purity can be obtained in arc melted ingots. The normal yield from electrode to conditioned EB melted ingot is reportedly about 90-95 percent.

The removal of volatile metals during EB melting imposes limitations on the types of alloys that can be melted using this process. Since it has been found that tungsten does not volatize during EB melting, the Ta-10W binary alloy can be successfully melted by this method. The ternary alloys, T-111, T-222 and the Ta-30Cb-7.5V alloy cannot be melted by this process because of the volatility of the vanadium and hafnium.

Table VI is a compilation of the melting data given in the replies to the questionnaire and previously reported[1] [10] [11].

INGOT EVALUATION AND CONDITIONING

To determine ingot soundness most of the companies contacted use a combination of visual, die penetrant and ultrasonic inspection. Prior to inspection the ingots are machine conditioned by removing between 1/16 inch and 1/4 inch from the surface. Surface finish of machine conditioned ingots is in the neighborhood of 125 RMS. All surface defects will be exposed in this manner. Die penetrant inspection methods are very good at revealing surface defects not visible to the naked eye. Internal defects are determined by ultrasonics. Both immersion and contact methods have been used. Allegheny Ludlum, as subcontractor to Wah Chang Corporation on Contract AF 33(600)-42396, ran a series of tests to determine the inspection technique giving the best resolution for Ta-10W alloy billets. The variables in this test were type and size of crystal and frequency. Both contact and immersion methods were evaluated. Instrument settings were standardized to give a two-inch screen indication from a 5/64-inch flat-bottom hole drilled 3/4 of an inch into the billet. It was reported[7] that the best results were obtained using the contact method and using a barium titanate 3/4-inch diameter crystal with a frequency of 5 mc.

Figures 6 and 7 show the surface conditions normally obtained on consumable arc melted and EB melted ingots. Figure 8 illustrates the large grain size obtained on as cast EB melted ingots. The ingots are cropped by sawing, abrasive cut-off or machine facing or a combination of these methods. The most effective method of surface conditioning is machining. Tantalum and its alloys machine similar to stainless steel. It has a tendency to gall and seize. Recommended machining practices are reported[12] as follows: High speed steel tools are preferred and should be used in conjunction with a lubricant. Recommended cutting speed is

TABLE VI (a)

Melting Data

Consumable Vacuum Arc Melting

Alloy	Electrode Dia.(in.)	Ingot Dia.(in.)	Voltage	Amps	Polarity	AC or DC	Furnace Atmosphere	Melt Rate (lbs/min.)
National Research Corporation								
Ta-10W	3	6	30	7000	Straight	DC	<1 micron	15
Ta-8W-2Hf (T-111)	3	6	30	7000	Straight	DC		
Stauffer Metals								
Ta-10W		8	30-32		Proprietary			
Ta-8W-2Hf (T-111)		8			Proprietary			
Wah Chang Corporation								
Ta-10W	5	8	30	14000		DC	Vacuum	15
Ta-30Cb-7.5V	6	9.5	35	17000		DC	Vacuum	18-25
Westinghouse								
Ta-8W-2Hf (T-111)	1st Melt		20-26	2400-3000		AC		3.3-3.7
	2nd Melt		30-32	3300		DC		3.3
T-222	1st Melt	1-7/8	28	2700		AC		2.5
	2nd Melt	3	30	3600		AC		5.5

TABLE VI (b)

Melting Data

Electron Beam Melting

Alloy	Ingot Dia.	Ingot Length	Power Requirements (KW)	Vacuum Pressure (microns)	Melt Rate (lb/min.)	Average Hardness (BHN)
National Research Corporation						
Ta-10W	2-1/4	30 in.	60	< .1	1/4	180
H. Starck Company, Goslar, Germany*						
Ta-10W	2 and 3	63 in.	23-28 Kwh/kg	.01-1	0.33	260
Wah Chang Corporation						
	5	40 in.	450	< .05	3	200 (3000 kg load)

*Information from H. Starck Company of Goslar, Germany, received from Shieldalloy Corporation.

Figure 6

Partially conditioned consumable
arc melted cast ingot of Ta-10W

(Photograph courtesy of National Research Corporation)

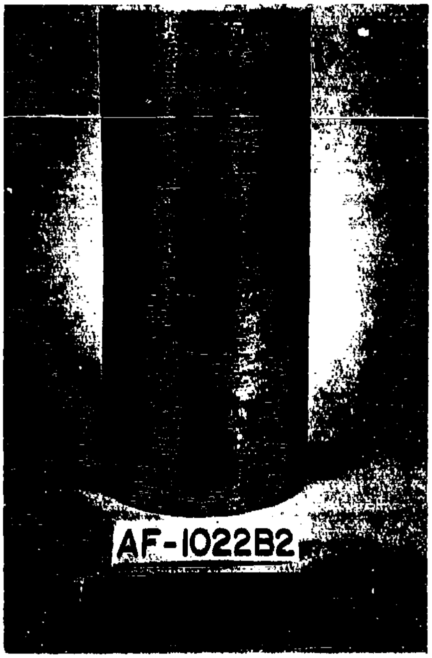


Figure 7

As-cast EB melted ingot of Ta-10W

(Photograph courtesy of Wah Chang Corporation)

Transverse

Longitudinal

Figure 8

Transverse and longitudinal macro structure of electron beam melted ingot.

(Bottom photograph courtesy of Wah Chang Corporation)

50-60 feet per minute with a roughing feed of .008-.012 inches per revolution. The depth of the cut should be between .015 and .060 inches at an approach angle of 30 degrees. Grinding of tantalum alloys is generally not practical. By careful machining and polishing finishes of 60 RMS or better are possible. Wah Chang reports ingot conditioning losses to be shrinkage 2-3 percent, surface conditioning 5-10 percent, hot top loss 3-5 percent, butt loss 1-2 percent, for an over-all average recovery of about 90 percent.

INITIAL INGOT BREAKDOWN

The initial breakdown of conditioned ingots can be accomplished by either direct forging or extrusion. Table VII lists the results of the current survey showing the fabricator, alloy composition and the methods of intial ingot breakdown used. Several known fabricators stated that they worked on a conversion basis and reported the fabrication details to be client confidential. Since the direct fabrication of compacted tantalum alloys has virtually ceased in the United States, this discussion includes only melted ingots. Also,the discussion is limited to those alloys which are under consideration as tubing alloys. A discussion of the forging and extrusion characteristics of experimental alloys is given in previous state-of-the-art surveys on tantalum alloys[1] [6].

Forging

All of currently commercial or semi-commercial tantalum alloys have been successfully forged using conventional equipment. Table VIII tabulates the forging procedures used by various companies on the various alloys. This table is a compilation of data reported in the current survey and data previously reported.

While it is possible to heat and work massive pieces of tantalum alloys bare in air, many fabricators pre-coat the ingots with glass frit or aluminide type coatings prior to heating. This helps prevent surface oxidation and greatly improves the yield. Another method of avoiding excessive oxidation is heating in a special retort containing a dynamic inert atmosphere. Most of the fabricators pre-heat the billets to temperatures in the range of 1800-2200F in a gas fired furnace with a reducing atmosphere. National Research Corporation reports[9] that breakdown forging of Ta-10W below 1800F, or continued working below 1500F may cause internal cracking.

To determine the effect of carbon content on the forgeability of Ta-10W, National Research Corporation prepared three Ta-10W ingots having carbon contents of 15, 60 and 120 ppm carbon. These ingots were upset forged at 2000F under identical conditions. They report[9] that the excess carbon drastically reduced forgeability and caused excessive edge cracking. Carbon contents of over 50 ppm are detrimental.

Torti[9] reports that 200-300 ppm of oxygen is sufficient to cause difficulty during initial breakdown of the Ta-10W alloy. Consequently, oxygen content is normally held to below 100 ppm and the total carbon, oxygen and nitrogen to less than 150 ppm.

Allegheny Ludlum, as subcontractor to Wah Chang Corporation on Contract AF33 (657)-7015, has forged two 7-inch diameter arc cast billets of the Ta-30Cb-7.5V alloy. The billets were coated in glass frit and heated to 2300F in a gas fired

TABLE VII

Methods of Initial Ingot Breakdown as Reported in Current Survey

Company	Alloy Composition	Method of Breakdown: Consumable Arc Melted	Method of Breakdown: EB Melted
Battelle	Ta-5W-2.5Mo	Extrusion	
	Ta-10W-2.5Mo	Extrusion	
Coulter	Ta-10W		Hammer Forge
Ladish	Ta-10W	Forge	Hot Forge
National Research Corp.	Ta-10W	Hot Forge	Hot Forge
Stauffer Metals	Ta-10W	Forging and Extrusion	Forging and Extrusion
Wah Chang Corp.	Ta-10W	Forge	Forge
	Ta-30Cb-7.5V	Forging and Extrusion	
	Ta-8W-2Hf	Forging and Extrusion	
Westinghouse	T-111 (Ta-8W-2Hf)	Forging and Extrusion	
	T-222 (Ta-9.6W-2.4 Hf-.01C)	Forging and Extrusion	

TABLE VIII

Forging Procedures Used for Initial Ingot Breakdown

Company	Alloy Composition	Billet Size	Forging Temp.(°F)	Type of Heating	Time to Temp.	Lubricant or Protective Medium Used
Allegheny Ludlum	Ta-30Cb-7V	7" dia.	2300	Gas Furnace		Glass
Coulter	Ta-10W	8" dia.	2150	Gas Furnace	75 min.	None
Ladish	Ta-10W	6" dia.-150 lbs.	2900-RT	Gas Furnace	30-45 min.	Glass Frit
National Research Corp.	Ta-10W	6" dia.	1800-2100	Argon-Muffler	1 hour	--
	Ta-8W-2Hf	6" dia.	1800-2100	Argon-Muffler	1 hour	--
Stauffer Metals	Ta-10W	Various	2200	Gas Furnace	Proprietary	
Wah Chang Corp.	Ta-10W	5" dia.	< 2000	Gas Furnace	20 min.	Glass
	Ta-30Cb-7.5V	8" dia.	< 2000	Gas Furnace	30 min.	Glass
	Ta-8W-2Hf	5" dia.	< 2000	Gas Furnace	20 min.	Glass
Westinghouse	T-111 (Ta-8W-2Hf)	3" dia.	2200	Gas Furnace	30 min.	--
	T-222	3" dia.	2200	Gas Furnace	30 min.	--

furnace with a reducing atmosphere. The billet was hammer swaged (closed die forging) to 5-inch round and subsequently forged on flat dies to 2-inch sheet bar. The first ingot was very stiff but forging was successful. The second ingot broke up during hammer swaging. Details of this work will be discussed in a forthcoming interim technical report on the above contract.

Ammon et al[11] report that the initial 3-inch diameter ingots of T-222 alloy were forged to plate in one heat from a temperature of 2200F. The billets were heated in a dynamic argon atmosphere and they were not given a protective coating of any kind.

Extrusion

Extrusion is another means of initial ingot breakdown. Experience and procedures used by the extruders for ingot breakdown of the various tantalum alloys are shown in Table IX. Successful extrusions have been accomplished in conventional, dynapak and impact extrusion presses.

Loewenstein describes[15] some of the techniques used in extrusion of refractory metal alloys. Streamlined flow must always be used. This can be achieved by using conical entry dies and conical approach billets. Entry angle should be in the range of 120-60 degree included angle. Streamlined flow can be obtained with flat die by using the Ugine-Sejournet glass lubrication process.

Billets as large as 8-1/2-inch diameter have been extruded. To achieve initial breakdown reduction ratios have generally been in the neighborhood of 4:1. Extrusion temperatures have varied between 2000 and 3100F, depending on the alloy, reduction ratio and other extrusion characteristics. Heating has been accomplished either in a salt bath or by induction in an argon atmosphere. This is necessary to keep oxidation of the billet surface to a minimum. Billets are often either clad in another metal such as carbon steel or molybdenum or coated with a glass slurry prior to heating. This aids both in eliminating surface oxidation and helps to overcome the problem of the tantalum alloy galling or sticking to the tools during extrusion. Ceramic coated tooling is another practice which aids in this respect.

Most of the extrusion experience has been with the Ta-10W alloy. On Contract AF33(600)-42396 a conditioned 8-inch diameter arc cast ingot was clad in steel and extruded at Canton Drop Forging and Manufacturing Company to a 4-1/4-inch diameter bar from a temperature of 2300F. Reduction ratio was 2.8:1. Figure 9 is a photograph of the declad extruded bar. Figure 10 is a photograph of an etched disc taken from the center of this extrusion. The grain structure is much finer than that found on cast ingots. This extrusion was subsequently machined into extrusion billets for use on the contract.

On this same Contract AF33(600)-42396, Allegheny Ludlum,as subcontractor to Wah Chang Corporation, has extruded both EB and arc cast billets of the Ta-10W alloy direct to a 1/4-inch "T" section. Billets were extruded from a 3-7/8-inch liner from temperatures in the range of 2700-3350F at a reduction ratio of about 18:1. Details of these extrusions are given in the fourth and fifth interim technical reports issued on this contract[13] [14].

Dupont reports[16] extruding 6-inch diameter ingots of both Ta-10W and T-111 at extrusion ratio of approximately 4.7:1 using glass lubrication. Billets were

TABLE IX

Extrusion Procedures Used for Initial Ingot Breakdown

Company	Alloy Composition	Billet Dia. (in.)	Reduction Ratio	Extrusion Temp. °F	Method of Heating	Time to Temp.	Heating Rate	Lubricant or Cladding	Max. Extrusion Pressure
Allegheny Ludlum	Ta-10W	3.875	18:1	2700-3350	Induction-Argon	3-15 min.	--	Proprietary Glass	Varied
Battelle*	Ta-5W-2.5Mo	3.88	4:1	2600	Induction-Argon	15 min.	--	Proprietary Glass	128,000 psi
Canton Drop Forging & Mfg. Co.	Ta-10W	8	2.8:1	2300	Salt Bath	--	--	Steel Clad	--
DuPont	Ta-10W	5.7	4.7:1	3100		--	--	Proprietary Glass	112,000 psi
	T-111	5.7	4.7:1	3050		--	--	Proprietary Glass	134,000 psi
Ladish	Ta-10W	3	4:1	2000	Gas Furnace	25-30 min.		Proprietary Glass	--
Wah Chang Corp.	Ta-10W	6.72	4:1	2300	Salt Bath	1 hour	--	Steel Clad	K=100,000 psi
	Ta-30Cb-7.5V	8.44	3.2:1	2300	Salt Bath	2 hours	--	Steel Clad	K=134,000 psi
	Ta-8W-2Hf	5.375	4:1	3000	Induction-Argon	15 min.	150°/min.	Proprietary Glass	K=50,000 psi
Westinghouse*	T-111 (Ta-8W-2Hf)	8	2.5-3.1:1	2800	Induction-Argon			Proprietary Glass	--

NOTE: *Battelle and Westinghouse reported extrusions were done at Dupont-Baltimore.

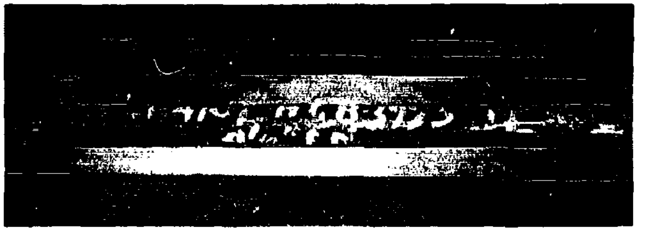


Figure 9

4-1/4-inch dia. declad extruded bar
of Ta-10W-extrusion temperature-2300F

Figure 10

Etched center disc from Ta-10W extrusion shown in Figure 9

heated and extruded bare; the Ta-10W from 3100F and the T-111 from 3050F. Billets were equipped with molybdenum nose pieces to lower the peak pressures and eliminate the unworked nose usually found in extrusions. This practice was also used by Allegheny Ludlum on Contract AF33(600)-42396.

Ammon et al of Westinghouse reports extruding from a 3-1/8-inch container on their 700-ton Lowey Hydropress as cast ingots of the T-111 alloy from a temperature of 3000F. Billets were plasma coated with molybdenum and extruded at ratios of 2.5:1 and 4:1. Molybdenum noses were used here also.

On Contract AF33(657)-7015 an 8-1/2-inch diameter conditioned ingot of Ta-30Cb-7.5V alloy was canned in carbon steel and extruded at Canton Drop Forging and Manufacturing to a 5-1/4-inch round from a temperature of 2300F. The extrusion was of an excellent quality although a sizeable discard was left.

Regardless of the method of ingot breakdown used it is imperative that the billets be thoroughly conditioned prior to subsequent working or heat treating. If this is not done surface oxides may diffuse throughout the billet on subsequent heating. Conditioning can be accomplished by grit blasting, machining, mechanical grinding and pickling. Quality of the forged or extruded pieces can be checked by die penetrant and ultrasonic inspection. If straightening of an extruded bar is necessary it can be done on a forging press at temperature between 1000 and 1800F.

FABRICATION OF TUBING

Although tantalum and tantalum alloy tubing is available in experimental amounts most of the users report that it has not been of a consistently high quality for use in nuclear aerospace applications. Tantalum and its alloys are quite ductile and work very readily by normal cold tube reducing and drawing processes. The main difficulty is in obtaining high quality tube blanks at a reasonable cost. Tube hollows have been made by a number of conventional and experimental processes. Some of the methods described in the literature are (1) drilling or tre-panning of bar stock (2) rolling sheet to a cylindrical shape and welding (3) cold extrusion (4) piercing and blanking (5) cupping either by impact extrusion or deep drawing (6) explosive forming (7) coating metal patterns and dissolving the pattern, and (8) hot extrusion. Most of the work reported in the literature was done on high purity tantalum or low alloys.

Until recently most producers of tantalum tubing had to depend on the drilling or tre-panning of bar stock to provide suitable tube hollows for subsequent tube reducing and drawing. Stock must be extremely straight to obtain a concentric tube hollow and it has a limitation on the length of the hollow provided. Recent developments in sheet rolling, welding and extrusion of tantalum alloys opened up many new methods of fabrication.

While the rolling of sheet to cylindrical shape and welding, a procedure commonly used in the steel and other metal industries, has been used for tantalum, it is generally agreed that welding techniques in refractory metals have not progressed to the degree of perfection required of quality tube blanks. It would also have a limitation on the thickness of the wall, since only thin sheet can be readily roll formed.

Quadt of Bridgeport Brass reports[17] [18] that high purity cast tantalum billets have been cold extruded into excellent quality tube blanks. By cold extrusion

the author means that the extrusion temperatures were below the recrystallization temperature of the metal and, in reactive metal such as tantalum, at temperatures below those at which the metal reacts with atmospheric gases. Hunter-Douglas Division of Bridgeport Brass has designed hydraulic presses ranging from 200-3500 tons for cold extrusion. Parts up to 20 inches diameter and 40 feet long have been cold extruded. The process, however, has thus far been unsuccessful with high strength alloys because of excessive tool loads. Lubrication is of prime importance in this process.

It has been reported[12] that blanking and piercing of tantalum alloys has been done on a press using highly polished steel punches and dies. Clearance between punch and die should be six percent of metal thickness. Equal parts of light oil and paraffin oil proved to be a good lubricant. However, this process has not been developed to the state required for commercial exploitation.

Closed end tantalum alloy tubes nine inches long with an ID of about 0.375 inch and a 0.020 to 0.030 inch wall are used as molten plutonium fuel containers in the LAMPRE I fast reactor experimental core facility. Two methods were used for fabrication of these containers: deep drawing the thimble from rolled sheet, and forming a heavy walled cup by impact extrusion of tantalum rod and subsequent ironing of the wall to form the desired thin walled container. Hanks et al [19] [20], have reported results of this development. A solid slug was given five impact steps to form a thick walled cup. The cup was then ironed through six stages to final shape. This is illustrated in Figure 11. Materials used were powder metallurgy tantalum, high purity EB melted tantalum and Ta-0.1%W alloy. Aluminum bronze dies and a cold beeswax lubricant were used to prevent galling between the tools and the tantalum. Beeswax coating should be very thin. The beeswax, starting slug and extrusion tools were kept at room temperature or below (cooled to about -30C). The investigation showed the Ta-0.1%W alloy consistently required greater pressures for both impact extrusion and drawing. Heat treating procedures greatly affected the surface of the finished tube. Recrystallized material consistently produced rougher surfaces than did material which was stress relieved without recrystallization. A stress relief treatment of one hour at 850C was given between the fourth and fifth impact extrusion steps.

Cashmore[21] of Accles and Pollock, Limited, reports producing tantalum tubing by the cupping and deep drawing technique. Sheets between .025 and .150 inch thick have been successfully cupped and deep drawn to a size suitable for conventional mandrel or plug drawing. Reductions in area in cupping passes are usually held to 45-50 percent calculated by the formula $\frac{Db - Dp}{Db}$ 100 were Db = blank diameter and Dp = punch diameter. For material thinner than .080 inch reductions should be limited to 22-23 percent based on the same formula. It is necessary to anneal during the deep drawing operation to prevent splitting. Tantalum alloy should be annealed after a 50-70 percent reduction in area. Anneal in vacuum 10^{-4} mm of mercury, 1200C, 15 minutes. Beeswax was again used as a lubricant during cupping and deep drawing.

Another method of making tantalum tubing is described in U. S. Patent 3,082,516[22]. This method consists of depositing the refractory material (tantalum or tantalum alloy) on a suitable metal support by heating and accelerating particles of this material in a stream of selected gas flowing through an electric arc while maintaining the temperature of such master support below its melting point. The resulting deposit layer has a characteristic microstructure in which microscopic lamellules or leaves of irregular shape are interlaced one with another. Inert

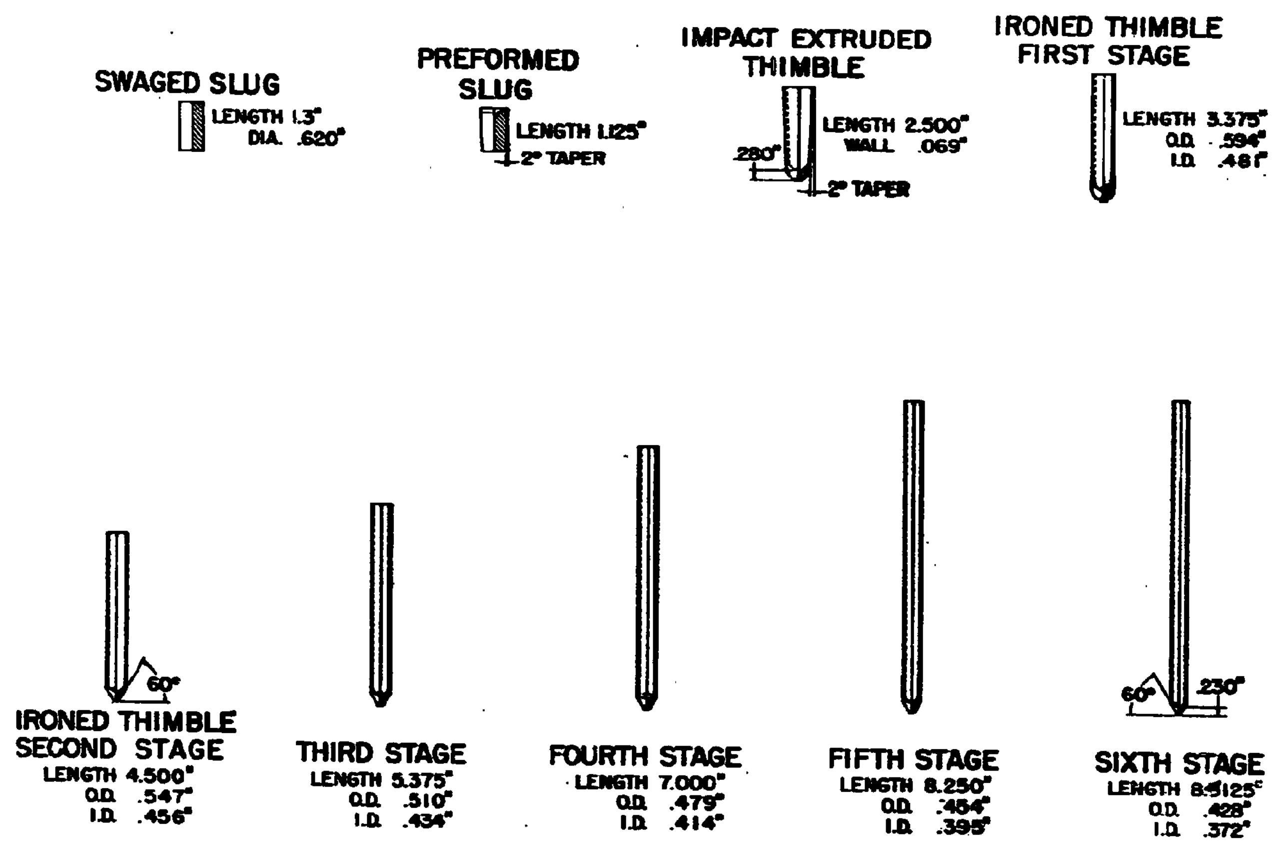


Figure 11

An illustration showing the final dimensions and cutaway views of the various stages during the impact extrusion and ironing process.

(Courtesy of Los Alamos Scientific Laboratory)

gases are usually employed to protect the refractory metal during the coating process. The master and shape are then separated from one another either by dissolving the master or mechanically removing.

Los Alamos Scientific Laboratory is currently making tantalum and columbium tubing 1/10 inch O.D. by .002 inch wall by 60 inches long by a vapor deposition method. Approximately $4,000,000 worth a year is required of this size for a classified application. They are making this at Los Alamos because the materials supplied by commercial producers and by the other methods could not meet the extremely high quality specifications.

In view of the recent developments in the hot extrusion of refractory metal alloys, it now appears that hot extrusion will offer the most economical and practical means of fabricating tube hollows for subsequent tube reducing or cold drawing. From one large billet multiple lengths of tube hollows can be obtained. Similarly tubes can be produced in a wide variety of sizes. This method would also offer the most promise in obtaining high quality tube hollows of high strength alloy, since many of the previously described methods have been successful only with high purity metals and low alloys. Allegheny Ludlum has successfully extruded 2-inch O.D. by .250 inch wall tube hollows of Ta-10W alloy from a 3-7/8 inch liner using extrusion techniques developed on Air Force Contract AF33(600)-42396. Successful extrusion of 2.250 inch O.D. by .375-inch wall tube hollows of this same alloy were accomplished from a 5-3/8 inch liner. Extrusion temperature in both cases was 3350F. Proprietary glasses were used as lubrication. Billets were heated by induction in an inert atmosphere.

In order to obtain tubing of the small diameters and thin walls required by most of the applications described in this report the tube hollows must be further reduced by a series of successive tube reducing, drawing and/or sinking operations to finished size.

To demonstrate the feasibility of producing tubing of the T-111 alloy, Westinghouse produced approximately eight feet of 3/8 inch O.D. by .035-inch wall tubing. In producing this a 3-inch diameter vacuum arc cast ingot was extruded to 1-7/8-inch diameter bar. This extrusion was machined into a tube hollow 1.5-inch O.D. by .220-inch wall. The tube blank was then tube reduced 73 percent at room temperature. All further processing was done at ambient temperatures with intermediate annealing of 2550F at a maximum pressure of 5×10^{-5} Torr.

Westinghouse Astronuclear Laboratory has an AEC Contract AT(30-1)3108 for the development of high strength columbium and tantalum alloy tubing. This program is outlined by Buckman[23] in the First Quarterly Report of this contract. The Westinghouse developed alloys B-66 and T-111 are the alloys selected for this program which require evaluation of three processing areas, (1) extrusion of tube blanks, (2) tube blank reduction to redraw stock, and (3) drawing to finished sizes. They are to produce tubing of the same sizes required in this contract, i.e., 1/2 and 3/8-inch O.D. by .062-inch wall and 1/4 O.D. by .020-inch wall. Tube blanks are to be extruded at Nuclear Metals, Inc. Billets are to be clad in mild steel and extruded from a 4.5-inch liner on a 1400-ton press. The tube blanks are to be tube reduced and cold drawn to finish size at Superior Tube Company.

J. Bishop and Company reports[24] drawing, swaging, and annealing thin walled tantalum tube in size ranges 0.009-inch O.D. by .002-inch wall to 1-inch O.D. by .008-inch wall. They report that to overcome cold welding of the tantalum to carbide dies an oxide conversion coating was used in conjunction with a

Bishop developed proprietary lubricant which can be applied directly at the draw bench. With this lubrication system reductions of area of 80 percent can be accomplished between anneals. Tubes were annealed and cooled in vacuum at 1850-1950F for 30 minutes.

Damascus Tube Company reports[25] producing tantalum tubes in the range of 1/2 to 2-3/4-inch O.D. by walls up to .148-inch by the "Flo-Rol" process. This is done on a machine manufactured by the Meta-Dynamics Division of Cincinnati Milling Machine Company. In this process tubes are placed on a mandrel and passed through a set of pulsating dies which gives a forge-like effect which removes minor surface irregularities and yields a finer grain structure than normal. Many internal contours can be produced by this process.

Both National Research Corporation and Superior Tube Company list Ta-10W and T-111 alloy tubing as regular mill products. National Research Corporation has produced sizes 0.03-1-inch O.D. by 0.0025-0.060-inch wall by lengths up to 20 feet. Superior Tube reports their size ranges as 0.012-inch O.D. by 0.0015-inch wall to 0.625-inch O.D. by 0.049-inch wall by 20 feet long. Figure 12 contains photographs showing two views of a Ta-10W tube 0.165-inch O.D. by 0.016-inch wall produced by Superior Tube Company. Figure 13 shows the microstructure of this same tube after a stress relief at 1800F.

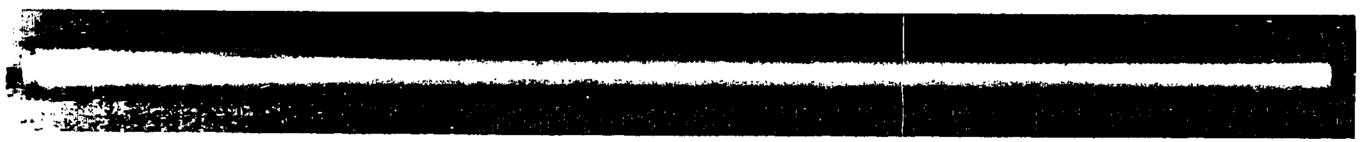
PROPERTIES OF TANTALUM TUBING ALLOYS

There is essentially no data available on the corrosion, physical or mechanical properties of tantalum alloy tubing. Until such time as sufficient tubing for a comprehensive testing program is available, it has been assumed that the properties will be comparable to those obtained for sheet products under similar test conditions. Since data on tantalum alloy sheets has been thoroughly covered in previous reports[1] [9] [10] [11] and in alloy data sheets issued by the producers of the alloys, only a brief resume' of these properties will be given in this report.

Corrosion Properties

As indicated in Table I the foremost property requirement for tantalum alloy tubing for use in nuclear aerospace applications is resistance to its environmental atmosphere, whether it be liquid alkali metals, liquid hydrogen or rocket exhaust gases. While it is general knowledge that tantalum and the other refractory metals display a unique corrosion resistance to liquid metals little long-time exposure data is available. NASA currently has some compatibility studies in process on thirteen commercially available columbium and tantalum base alloys. This work is being carried out at both the Lewis Research Laboratory in Cleveland and by other companies or government agencies working in conjunction with NASA. It is known that good compatibility is dependent upon the purity of the liquid metal. Intergranular attack is evident if oxygen is present in the liquid metal. This is especially true in the case of Na or NaK, where most of the testing to date has been done. It is hoped that sufficient 1000-hour corrosion data will be available by December, 1963, or early 1964 to make some decisions as to alloy selection.

At Los Alamos Scientific Laboratory tantalum tubing is used as fuel capsules to contain the liquid plutonium fuel for the LAMPRE I reactor. Liquid sodium is used as the coolant in this reactor. Accordingly these tubes must be



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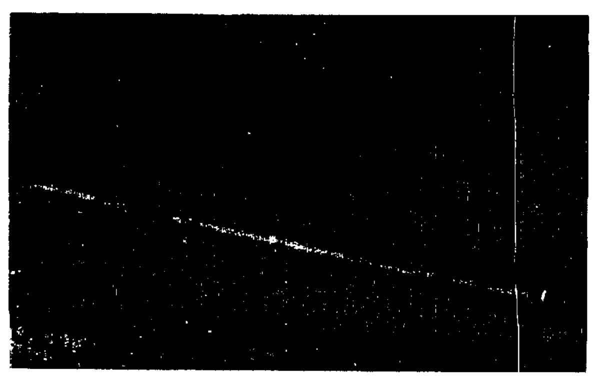

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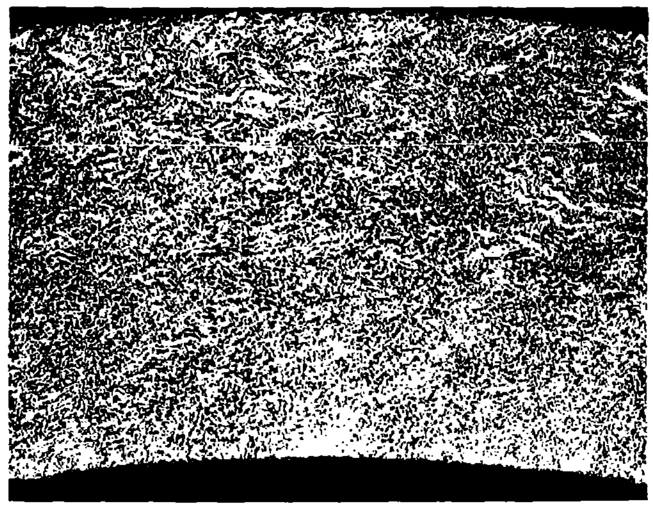
Figure 12

Photographs showing two views of a Ta-10W tube 0.165-inch O.D. x 0.016-inch wall



Mag. 100X

Figure 13

Ta-10W Tubing .165-inch O.D. x .016-inch wall
Transverse - 1800F - Stress Relief
Etchant NH_4F-HF

(Photograph Courtesy of Superior Tube Company)

corrosion resistant to both liquid plutonium and liquid sodium. They are currently running a series of tests. Tests are run in a natural convection sodium loop at temperatures of 650-700C. To determine if liquid plutonium has penetrated the wall of the capsule an alpha sensitive autoradiographic film is wrapped around the capsule. Penetration will be observed as spots on the film. The size of the spots, when present, is in the order of microns. Penetration has been observed anywhere from 200 to several thousands of hours. Despite evidence of penetration no weight gain or loss has been observed. Plutonium evidently runs down the grain boundaries and leaks out. Some evidence of attack by liquid metals is observed in the weld heat affected zone. Metallographic examination has revealed 1/2 to 1 grain deep platelets in the weld area.

Early tests on the Ta-0.1W alloy revealed that this alloy was poorest corrosion-wise. Tungsten inclusions were found in the early heats and these were believed to be the cause of the poorer corrosion resistance. By going to double arc melting they were able to homogenize the tungsten, and recent heats of the alloy are comparable to the pure tantalum.

Tests are also being run at Lewis Laboratory of NASA on the corrosion of unalloyed tantalum in hydrogen atmosphere. The test consists of heating tantalum strip in an atmosphere of dry purified hydrogen. A gradient of temperatures from room temperature to 3000F is obtained in the strip. If the piece is heated, and held at temperature one hour and cooled in the hydrogen atmosphere it was found that the tantalum falls apart in the area heated to 2000-2500F. This does not occur if the specimen is heated and cooled in argon but held for one hour at temperature in a hydrogen atmosphere.

Since space is an almost perfect vacuum one of the serious shortcomings of tantalum alloys, namely poor oxidation resistance at elevated temperatures is of little concern in aerospace applications.

Weldability

One of the primary prerequisites in many of the applications for tantalum alloy tubing is the ability to produce sound stable welds. Tantalum and tantalum alloys have been successfully welded to themselves and to other metals. Simple TIG (i.e., tungsten-inert-gas) welding using either butt welding or filler rod techniques is the most common practice. Other methods which have been successfully used on tantalum tubing are (1) high-frequency resistance pressure welding, EB welding and plasma arc welding. To achieve sound ductile welds by any of these processes the preparation of materials and welding procedures must be kept under rigid control. Los Alamos has indicated that surface films on the metals give trouble during welding. Even pickling just prior to welding is not sufficient. They find it necessary to pickle and abrade the surface just prior to welding. They also noticed sudden explosions occurring while welding. These, they believe, are due to inclusions in the base metal.

It was reported in an earlier state-of-the-art survey (1) that some loss of room temperature tensile ductility was encountered when Ta-10W sheet was welded using the TIG welding technique with a Ta-10W filler rod. However, no loss in strength or ductility of the Ta-10W weld was encountered when the electron beam welding technique was employed. Tests on the Ta-30Cb-7.5V alloy using TIG welding without a filler rod showed that the excellent room temperature bend ductility was maintained while tensile ductilities and strengths were slightly reduced. These results are shown in Table X.

TABLE X

Weldability of Tantalum Alloys

Composition	Joining Method	Tensile Properties UTS 1000 psi	TYS 1000 psi	Elongation %	Minimum Bend Radius T
Ta-10W	Base Metal	90	83.5	28.7	< 1
	TIG + Ta-10W Filler	87	75	9	--
	Base Metal	91	83.5	28.5	< 1
	EB Weld	91	81.5	26.5	--
Ta-30Cb-7.5V	Base Metal	120	104	29	0
	TIG-No Filler	116	92.5	19	0

Stauffer reports that STA-900 (EB melted Ta-10W alloy) can be welded by electron beam welding or inert gas fusion welding. Starting material should be stress relieved or preferably fully recrystallized. They report that if properly welded the properties are essentially the same as those of the base metal.

Ammon and Begley of Westinghouse[10] [11] report the results of weld bend ductility tests on the T-111 and T-222 alloys and a weld aging study on the T-111 alloy. Material was automatically TIG butt welded in a vacuum chamber back filled with argon. Sheet stock 0.050 to 0.060-inch thick was rigidly held in a copper clamping device designed to give high restraint while providing a heat sink for rapid cooling of the welded material. Specimens of both stress relieved and recrystallized material were tested in the as-welded condition. The T-111 alloy was tested at -320F at a deflection rate of 1-inch per minute over 2T and 4T bend radius. T-111 specimens were tested with the weld running parallel to the bend axis and with the weld running parallel to the long dimension. Results of both type tests showed that at -320F welded T-111 sheet with base metal in either the stress relieved or recrystallized condition was completely ductile over 2T and 4T bend radii. T-222 specimens were tested with the weld parallel to the long dimension of the specimen using a mandrel with a 2.5T bend radius. Tests were run at -250F and -320F. The weld specimens tested at -250F exhibited ductile behavior for both the stress relieved and the recrystallized base metal. At -320F failure was encountered in the welds of both specimens with stress relieved and recrystallized base material. The transition temperature for ductile to brittle behavior in welded T-222 appears to be between -250 and -320F for the 2.5T bend condition.

The weld aging study on T-111 sheet was run to determine the stability of the base metal, fusion and heat affected zones after one hour aging treatments at temperatures from 1200 to 2200F. The hardness surveys showed no gross hardness variation after aging at all temperatures. Metallographic study showed that no second phase appears in the weld metal or heat affected zone after aging for one hour at temperatures up to 2200F.

Physical and Thermal Properties

Available density, melting point and room temperature electrical resistivity data for some of the leading tantalum tubing alloy candidates is given in Table XI. Figure 14 is a plot of electrical resistivity of unalloyed tantalum and the T-111 alloy over a temperature range of -320F to 2750F. Table XII includes the average coefficient of thermal expansion for the Ta-10W alloy and the T-111 alloy in temperature range of 80-4350F.

Stress Relief and Recrystallization Behavior

Annealing operations for all tantalum alloys must be conducted in either a vacuum or inert atmosphere. The recrystallization range for any alloy is naturally dependent upon the amount of work in the material and the time at temperature. Table XIII shows the stress relief and recrystallization anneals recommended by the various producers of tantalum alloys.

Torti[9] states that recrystallization of Ta-10W after initial breakdown should be carried out at 3000F for one hour. However, if material has received more than 80 percent cold reduction in area an anneal of 2800F for one hour will give an even, fine grained structure of ASTM No. 9 grain size. Wah Chang[27] gives a

TABLE XI

Density, Melting Point and Room Temperature Electrical Resistivity of Tantalum Alloys

Composition	Density lb/in^3	Melting Point °F	Electrical Resistivity at RT - Microhom - CM
Ta	0.600	5420	12.5
Ta-10W	0.608	5516	18
STA-900*	0.608	5495	--
Ta-8W-2Hf	0.604	5400	22
Ta-30Cb-7.5V	0.426	4405 ± 90	

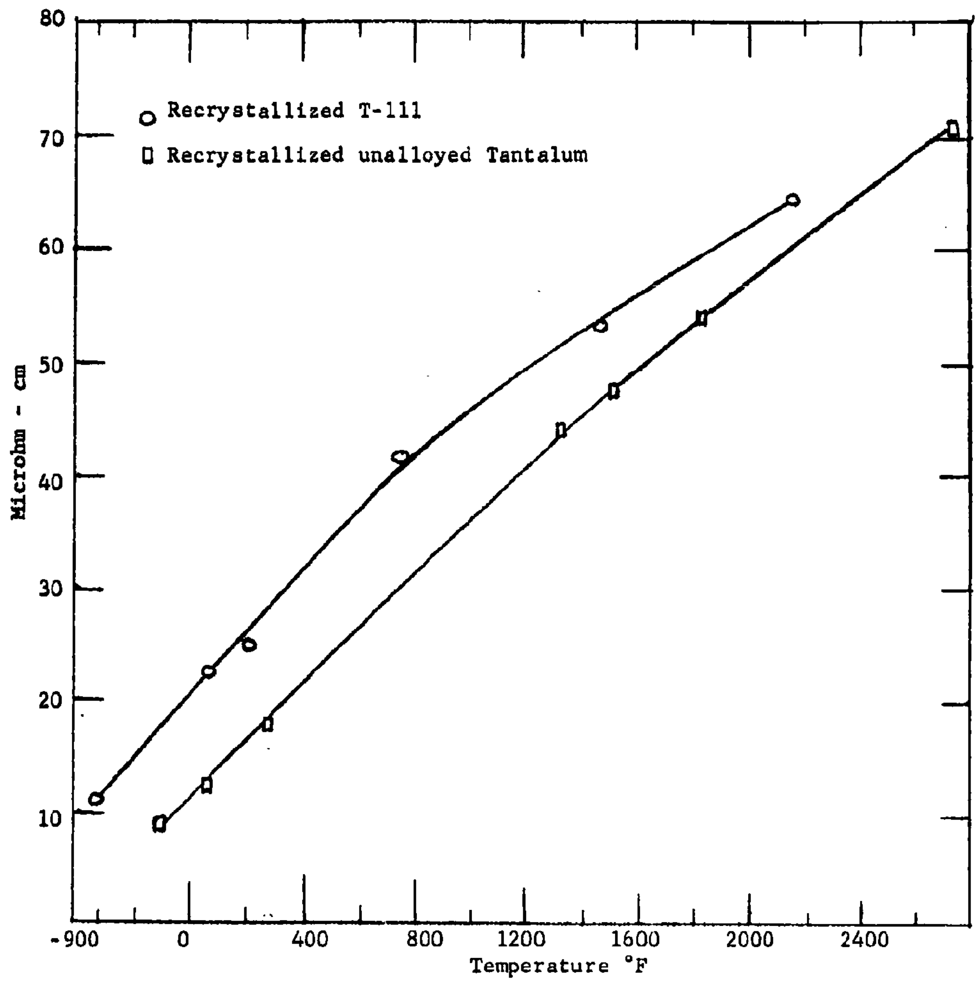


Figure 14

Electrical Resistivity of Tantalum and T-111

TABLE XII

Average Coefficient of Thermal Expansion for the Ta-10W and T-111 Alloys (1) (10)

Temperature °F	Average Coefficient in/in/°F x 10^6 Ta-10W	T-111
80- 500		3.1
80-1000		3.5
80-1500		3.9
80-1830	3.1	
80-2000		3.9
80-2010	3.2	
80-2190	3.3	
80-2370	3.4	
80-2500		4.0
80-2550	3.6	
80-2730	3.7	
80-2910	3.8	
80-3000		4.2
80-3090	3.85	
80-3270	4.0	
80-3450	4.1	
80-3500		4.2
80-3630	4.2	
80-3810	4.25	
80-3990	4.3	
80-4000		4.2
80-4170	4.4	
80-4350	4.5	4.3

Values for Ta-10W alloy were converted from $\Delta L/L$ to in/in/°F x 10^6

TABLE XIII

Recommended Stress Relief and Recrystallization Annealing Treatments

Company	Alloy	Stress Relief			Recrystallization			
		Temp. °F	Time	Atmosphere	Temp. °F	Time	Atmosphere	Recrystallization
Wolverine	Unalloyed Ta	1800	1 hour	Vacuum 10^{-5} Torr	2200	1 hour	10^{-5} Torr	100%
Westinghouse	T-111	2000	1 hour	Vacuum 5×10^{-6} Torr	3000	1 hour	5×10^{-6} Torr	100%
	T-222	2000	1 hour	Vacuum 5×10^{-6} Torr	3000	1 hour	5×10^{-6} Torr	100%
Wah Chang Corp.	Ta-10W	2000	1 hour	Vacuum	2400	4 hours	Vacuum	>80%
	T-111	2000	1 hour	Vacuum	2400	4 hours	Vacuum	>80%
	Ta-30Cb-7.5V	2000	1 hour	Vacuum	2200	1.5-2 hours	Vacuum	>80%
National Research	Ta-10W	2150	3 hours	Vacuum <.1 micron	3000	1 hour	Vacuum	100%
					3000	1 hour	<.1 micron	
Stauffer Metals	STA-900 (Ta-10W)				2600	1 hour	Vacuum	100%

recrystallization temperature range of 2500 to 2800F and has indicated that 80 percent recrystallization is obtained on Ta-10W with an anneal of four hours at 2400F. Stauffer[26] indicates that 100 percent recrystallization of their STA-900 (EB melted Ta-10W) alloy can be achieved by heating for one hour at 2600F regardless of the amount of cold work in the material. Figure 15 shows representative wrought and fully recrystallized grain structures of the Ta-10W alloy.

Ammon and Begley[10] report that, as would be expected, T-111 alloy material which received lesser amounts of cold work require higher temperatures to initiate and complete the recrystallization process. From their data it appears that for material cold worked 90 percent or more a temperature of about 2550F would be required to produce a 50 percent recrystallized structure in one hour. Their data also indicated a strong dependency of the hardness of recrystallized material on the amount of cold work received prior to recrystallization. A similar behavior was noted at Battelle for the Ta-30Cb-7.5V alloy. This was reported in the state-of-the-art analysis in Contract AF33(657)-7015[1]. Figure 16 illustrates the effect of annealing temperature on the room temperature hardness of the various alloys. Ammon and Begley[10] data also indicated that the amount of cold work prior to recrystallization had a significant effect on tensile properties at 2400F. The material with the lower amount of cold work prior to recrystallization displayed the higher tensile properties.

Recrystallization behavior of the T-222 alloy is currently being studied at Westinghouse Astronuclear Laboratory under Navy Contract N600(19)-59762. One hour at 3000F will completely recrystallize the alloy.

Mechanical Property Evaluation

Published data on the mechanical properties of tantalum alloy tubing is non-existant. Most of the preliminary alloy evaluation for tubing applications have and are being run using tantalum alloy sheet material. It is assumed that the data for tubular products will be comparable to the data accumulated on sheet material having a similar fabrication and under similar test conditions. Therefore, a brief resume' of previously reported mechanical property data on tantalum alloy sheet material is given here.

Room Temperature Tensile Properties

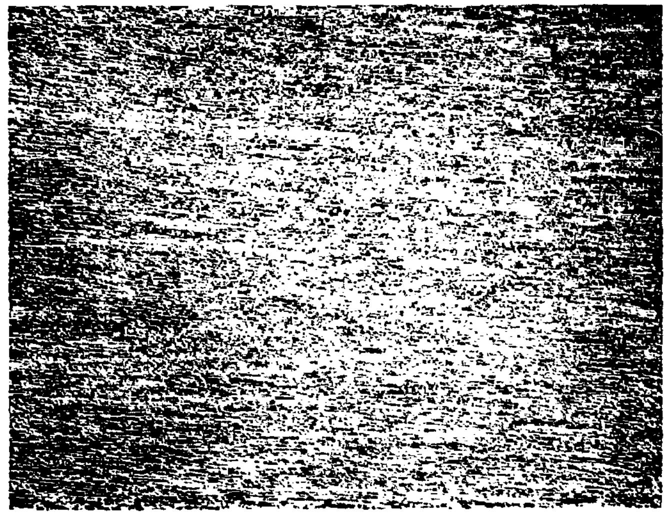
Room temperature tensile data for cold rolled Ta-10W sheet with 95 percent reduction is given in Table XIV. The data shown was obtained from three sources and the tests were conducted on material in both the as rolled and annealed conditions.

Table XV shows the room temperature tensile properties for cold rolled T-111 (Ta-8W-2Hf). Material was tested parallel to the rolling direction.

Room temperature tensile data in the Ta-30Cb-7.5V alloy as reported[1] by Battelle are given below:

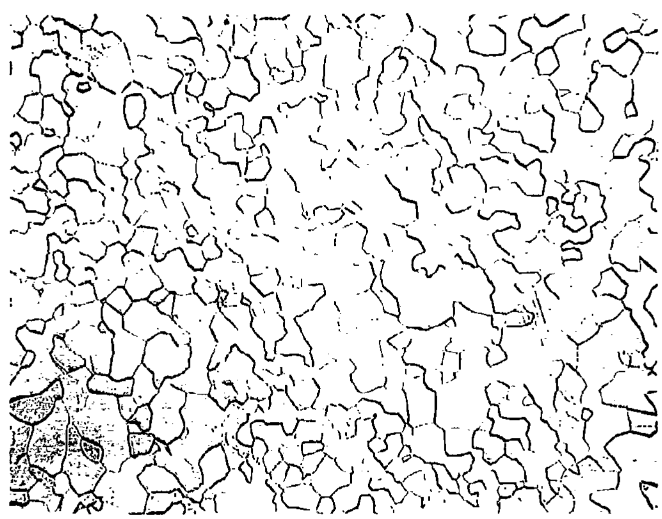
	Stress Relieved 1/2 hour at 1800F	Recrystallized 1 hour at 2200F
Ultimate Strength, ksi	154.5	120
Yield Strength, 0.2% offset, ksi	139	105
Elongation, %	17	28.5

Above data is the average of two tests both tested using strain rate of 0.02 inch per inch per minute.



Mag. 100X

Ta-10W - 1/2-inch dia. cold swaged to 1/4-inch dia.
Etchant HNO_2 - H_2SO_4 - HF



Mag. 100X

Ta-10W - 1/2-inch dia. swaged to 1/4-inch dia. and
Vacuum Annealed 4 min. @ 3200F
Etchant - HNO_3 - H_2SO_4 - HF

Figure 15

Representative grain structure of Ta-10W in
the Wrought and fully recrystallized condition

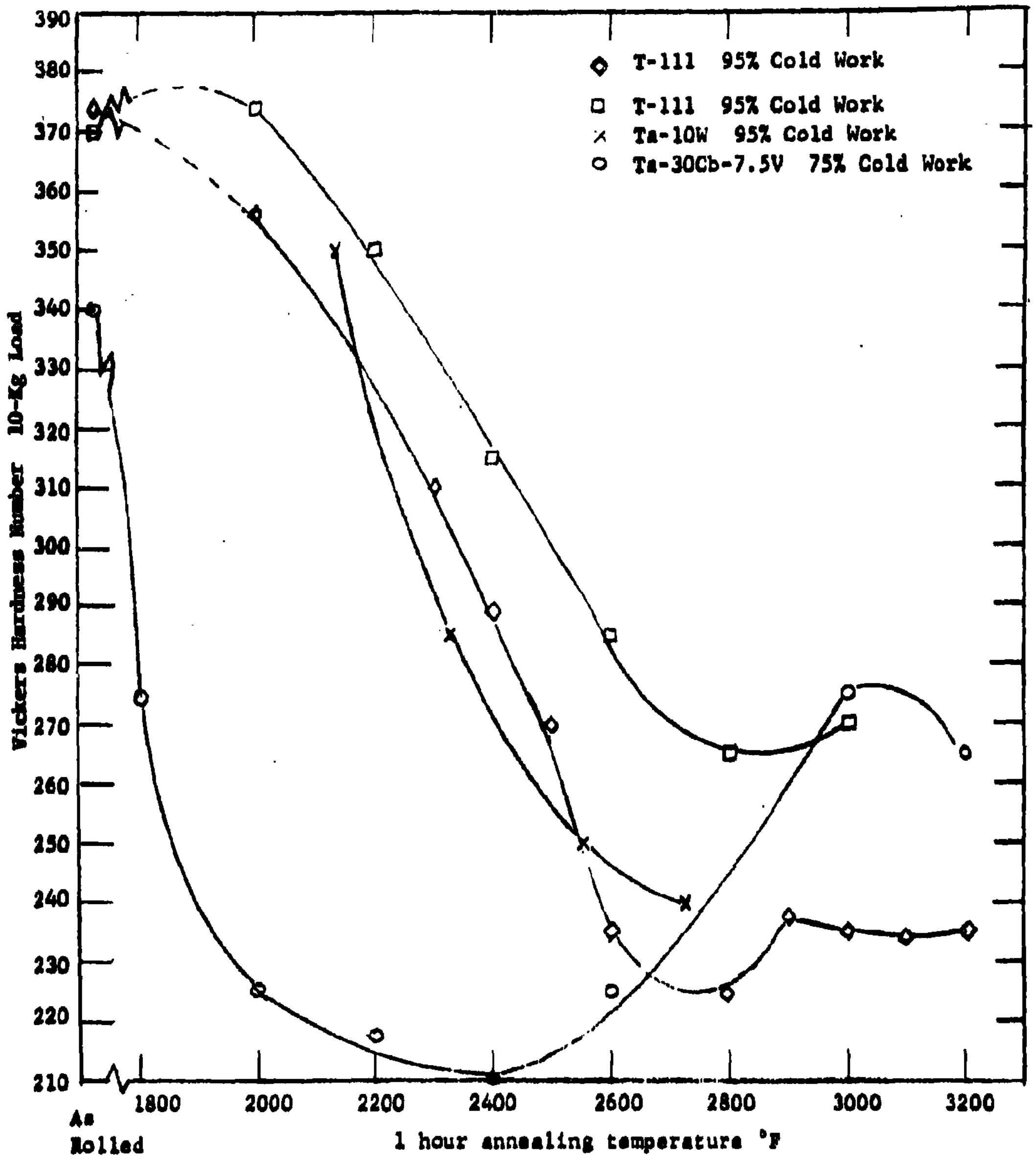


Figure 16

Effect of Annealing Temperature on the Room Temperature Hardness of Various Tantalum Alloys

TABLE XIV

Room Temperature Tensile Properties
Ta-10W Alloy Sheet

Source	Condition	Ultimate Strength psi	Yield Strength 0.2% Offset psi	Elongation %
A	As Cold Rolled	180,000	164,000	4.0
	Stress Relieved (3 hrs @ 2150F)	121,000	108,000	15.0
B	As Cold Rolled	180,000	160,000	4.0
	Annealed 4 hrs @ 2400F	95,000	81,000	35.0
C	Annealed 3 hrs @ 2200F	112,000	109,000	12.0
	Annealed above 2500F	80,000	67,000	25.0

TABLE XV

Room Temperature Tensile Properties of T-111[a]

Condition	Tensile Strength (psi)	Yield Strength 0.2% Offset psi	Elongation %
As Rolled	167,200	159,600	4.4
Annealed 1 hr @ 2000F	131,000	125,000	12.8
Annealed 1 hr @ 2200F	122,000	115,000	16.9
Annealed 1 hr @ 2400F	91,300	88,900	29.1
Annealed 1 hr @ 2700F	91,900	90,400	30.8
Annealed 1 hr @ 3000F	99,800	87,300	28.4
Annealed 3 hrs @ 2250F[b]	112,300	96,000	21.8

(a) Data from Westinghouse Data Sheet on T-111 Alloy.

(b) Data from National Research Corp. Data Sheet on .040-inch thick sheet.

Only one 22-pound ingot of the T-222 (Ta-9.6W-2.4Hf-0.01C) alloy has been melted and processed to date. Room temperature tensile data as reported by Westinghouse[11] for material which was reduced 90 percent and recrystallized one hour at 3000F is:

Ultimate Strength, ksi	110.0
Yield Strength, 0.2% offset, ksi	105.0
Elongation, %	25

Notched Tensile Properties

Battelle, on Contract AF33(616)-7604, tested a series of notched and unnotched tensile specimens of the Ta-10W alloy in both the annealed and wrought condition. Specimens were tested in the temperature range of -434F to 75F. The data presented in Table XVI shows that the alloy retains good tensile ductility at temperatures as low as -434F. The notch-unnotch strength ratio was in all cases about 1.5.

Westinghouse performed notch tensile tests at room temperature and -320F for the T-111 alloy. The sheet was reduced 90 percent and given a recrystallization anneal of one hour at 3000F. The data[10] are listed in Table XVII. The notched-unnotched strength ratio for the T-111 alloy was 1.15.

Elevated Temperature Tensile Properties

Elevated temperature tensile properties for the Ta-10W alloy as reported by Wah Chang are given in Table XVIII. Torti[9] notes that the strain rate has a large effect upon the strength at elevated temperatures. The results of tests run at National Research Corporation at various strain rates are shown in Table XIX.

Elevated temperature tensile data for the T-111 alloy is presented in Table XX. Data presented is taken from two sources. High elongations were found for materials tested at 2700F in the wrought stress relieved condition. Ammon and Begley[10] explain this as follows: Since the test temperature was just above the recrystallization temperature for this material, a fine grain size was produced which was apparently able to accommodate more plastic strain. As test temperature was raised, longer heating times as well as higher temperature, promoted grain growth, thus resulting in a return to normal ductility values for the material tested well above the recrystallization temperature.

Elevated temperature data on the T-222 alloy as reported by Ammon and Begley[11] is given below:

	Test Temperature °F		
	2400	3000	3500
Ultimate Strength, ksi	53.4	24.9	14.2
Yield Strength, 0.2% offset, ksi	37.8	24.1	14.2
Elongation, %	20	24	43

This material was reduced 90 percent and had received a recrystallizing anneal of one hour at 3000F.

Table XXI contains the elevated temperature data determined by Battelle and NASA for the Ta-30Cb-7.5V alloy.

TABLE XVI

Tensile and Notch Tensile Properties of Ta-10W

Material	Specimen	Test Temp (°F)	Ultimate Strength (1000 psi)	Reduction in Area (%)
Wrought	Notched	75	190	85
	Unnotched	75	130	85
	Notched	-100	225	85
	Unnotched	-100	160	85
	Notched	-325	290	70
	Unnotched	-325	210	75
	Notched	-434	350	55
	Unnotched	-434	235	70
Annealed	Notched	75	135	90
	Unnotched	75	85	95
	Notched	-100	170	85
	Unnotched	-100	115	88
	Notched	-325	225	60
	Unnotched	-325	160	70
	Notched	-434	290	25
	Unnotched	-434	180	65

TABLE XVII

Notched Tensile Properties of T-111

Condition	Temp. °F	Ultimate Strength (ksi)	Yield Strength 0.2% Offset (ksi)	Elongation (%)	Reduction In Area (%)
Notched	75	96.85		7	82
Unnotched	75	85.2	75.4	40	70
Notched	-320	157.7		5	32
Unnotched	-320	136.5	127.4	34	67

TABLE XVIII[27]

Elevated Temperature Tensile Properties of Ta-10W

Temp. °F	Ultimate Tensile Strength 1000 psi	Yield Strength 0.2% Offset 1000 psi	Modulus of Elasticity 10^6 psi	Strength to Wt. Ratio, 1000 psi/lb/in^3
1500	103.2	97.8	10.6	170
2500	22.25	19.85	8.55	36.7
3000	12.1	11.8	9.25	19.9
3500	7.48	7.26	7.85	12.3
4000	4.35	4.30	5.75	7.17
4500	2.06	2.06	2.50	3.39
5000	0.645	0.645	0.785	1.06

TABLE XIX[9]

Elevated Temperature of Ta-10W
Tensile Properties at Varying Strain Rates
.060-inch Thick Sheet
Cold Rolled
95 Percent Reduction in Area

	Fast Load Rate (a)			Slow Strain Rate		
Temp. °F	U.T.S.	Y.S.	% Elong.	U.T.S.	Y.S.	% Elong.
		1000 psi			1000 psi	
1500				(b)103	98	11
1800				(c) 94	80	4
2200				(c) 67	55	4
2500	58	44	3	(b) 22	20	22
2600				(c) 21	14	17
3000	25	15	13	(b) 12	12	33
3500	16	11	24	(b) 7.5	7.2	37
4000				(b) 4.3	4.3	35
4500	7.5	3.5	14	(b) 2.1	2.0	30
5000				(b) .6	.6	20

Notes:

All specimens heated by self-resistance in argon.

(a) Tested at Aerojet-General, Azusa, California
Loading rates 12,400 psi/sec. at 2500F, 4500 psi/sec. at 3000F, 1950 psi/sec. at 3500F and 4500F.

(b) Tested at Southern Research Institute, .01 in/in/min.

(c) Tested at Marquardt Corporation, .001 in/in/sec. to yield, .01 in/in/sec. to fracture.

TABLE XX

Elevated Temperature Tensile Properties of T-111 Sheet

Temp. °F	Ultimate Strength (ksi)	Yield Strength 0.2% Offset (ksi)	Elongation (%)	Strain Rate (in/in/min)	Remarks
2200	85	78	15	0.06	95% Reduction
2400	57.6	49.5	24.7	0.06	Stress Relieved
2500	54	38	26	0.06	1 hour @ 2000F
2700	29	23.7	64	0.06	Specimen -
3000	20.5	19.5	60	0.06	0.050-in. thick
3000	17.2	17.2	46	0.05	Recrystallized 1 hour @ 3000F
2000	92.1	67.5	8	0.05	90% Reduction
2200	67.1	52.2	20	0.05	Stress Relieved
2400	42.4	38.6	28	0.05	1 hour @ 2000F
2700	25.4	21.0	76	0.05	Specimen -
3000	16.3	14.1	52	0.05	0.028-in. thick
3500	11.3	10.9	43	0.05	
2400	37.3	23.5	36	0.05	Recrystallized
3000	14.8	11.9	48	0.05	1 hour @ 3000F
3500	13.0	12.6	34	0.05	
2000 (a)	67.6	65.2	4	0.05	95% Reduction
2400 (b)	40.5	33.1	41.7	0.05	Stress Relieved
2500 (a)	37.0	32.0	13	0.05	3 hours @ 2250F
3000 (a)	20.3	17.9	29	0.05	Specimen -
3500 (a)	11.2	11.2	31	0.05	0.040-in. thick
4000 (a)	4.25	3.97	33	0.05	

(a) Tested at Southern Research Institute

(b) Average of three tests run at Watertown Arsenal under MAB sponsorship

TABLE XXI

Elevated Temperature Tensile Properties of Ta-30Cb-7.5V Alloy Sheet

Temp. °F	Ultimate Strength (ksi)	Yield Strength 0.2% Offset (ksi)	Elongation (%)	Source of Data
	Stress Relief Anneal 1/2 hour @ 1800F			
1000	138	117.5	11	Battelle
1800	87	41	32	Battelle
2200*	42.5	32	64	Battelle
	Recrystallized 1 hour at 2200F			
1000	104.5	69	20	Battelle
1800	76	53	38	Battelle
2200*	46	33.5	50.5	Battelle
2400	33	26.7	87	Battelle
2700	19	16.2	>100	Battelle
3000*	10.5	8.6	>100	Battelle
3300*	6.6	6.6	>100	NASA
3500	4.4	-	>100	NASA
3750	2.7	-	>100	NASA
3980	1.6	-	>100	NASA

*Average of two tests

NOTE: Tests at Battelle - Strain Rate 0.02 in/in/min

Tests at NASA - Strain Rate 0.05 in/in/min

Stauffers elevated temperature tensile data for their STA-900 alloy is presented in Figure 17.

Figure 18 is a plot of the ultimate tensile strength/density ratio versus temperature for the Ta-10W, Ta-30Cb-7.5V and T-111 alloys.

Stress Rupture Properties

Stress rupture properties of the Ta-10W alloy, including Stauffer's STA-900 at test temperatures from 2000 to 4800F, are summarized in Figure 19. Data presented is from three different sources: Wah Chang, Stauffer Metals and National Research Corporation.

Westinghouse has reported[10] stress rupture properties of T-111. Specimens were tested at 2400F in vacuum at pressures below 5×10^{-5} Torr. Samples were tested in both the stress relieved and fully recrystallized condition. The data is presented in Table XXII. The recrystallized samples exhibited somewhat higher rupture times and lower creep rates at equivalent stress levels than the stress relieved material.

Stress rupture data is not available on the Ta-30Cb-7.5V and the T-222 alloys.

For true evaluation of the alloys for nuclear aerospace application 10,000-hour creep data is greatly needed, since it is felt that failure will occur by bulging and longitudinal creep. NASA and their affiliates are currently running comprehensive creep tests on all the alloys under consideration. Creep data to 1000-hours should be available by December, 1963.

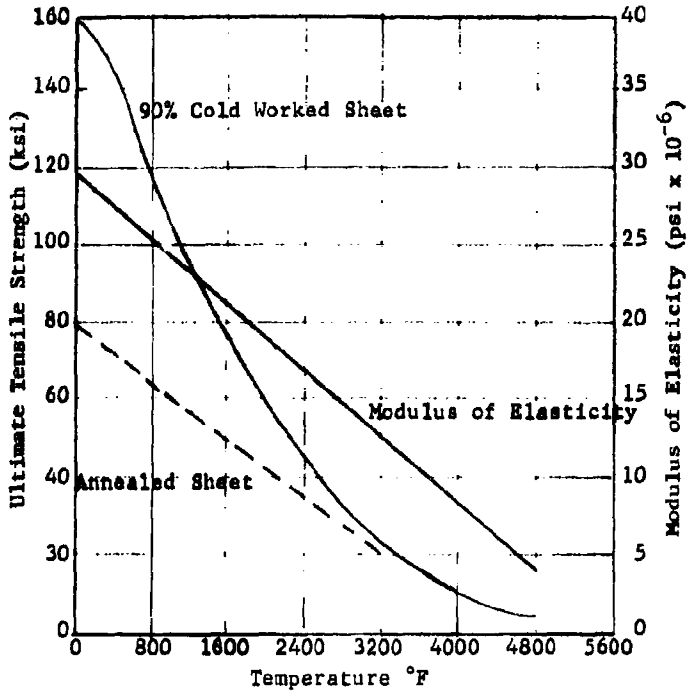


Figure 17

Elevated Temperature Tensile Data
for the Stauffer Ta-10W Alloy STA-900

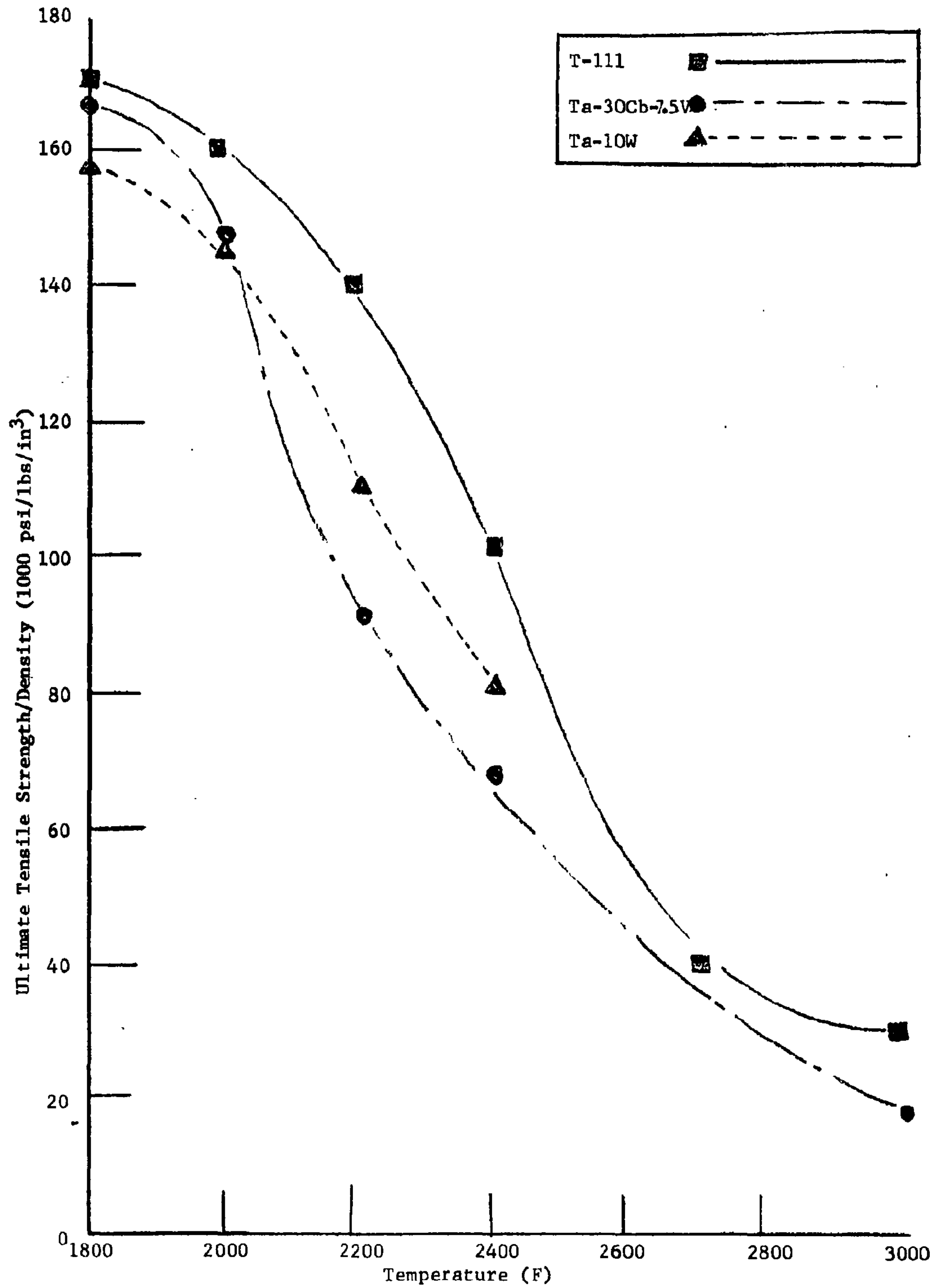


FIGURE 18

ULTIMATE TENSILE STRENGTH/DENSITY RATIO VS.
TEMPERATURE FOR SELECTED TANTALUM BASE ALLOYS

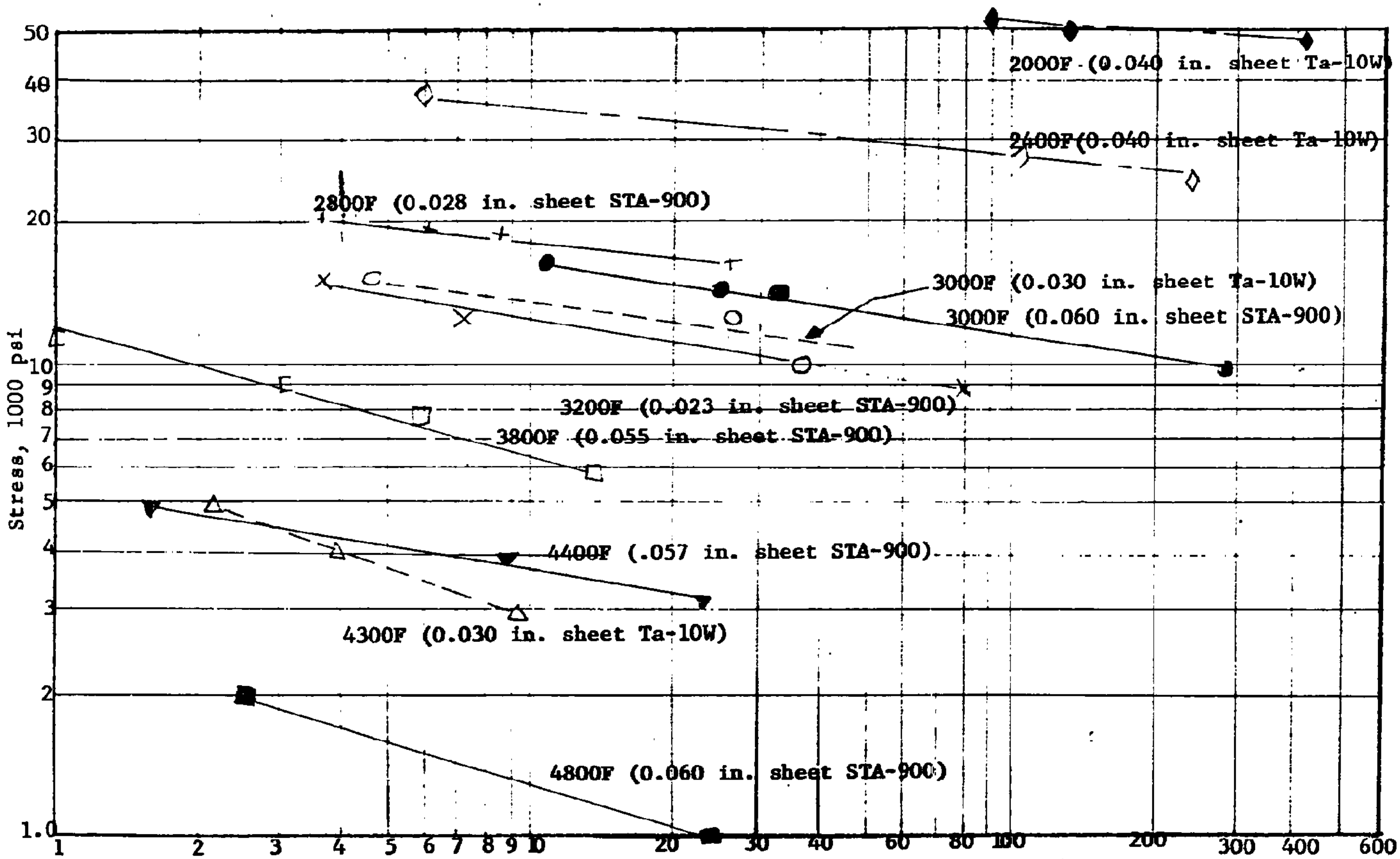


Figure 19

Stress Rupture Strengths of Ta-10W and STA-900 (EB melted Ta-10W) at Elevated Temperatures

TABLE XXII[10]

Stress Rupture Properties of T-111 Sheet Tested at 2400F

Stress ksi	Minimum Creep Rate %/hr	Transition Time (hrs)	Rupture Time (hrs)	Elongation (%)	Remarks
30	5.87	2.44	3.0	30	Material Reduced
25	1.28	2.25	7.3	40	80% -
25	0.34	12.0	27.0	21	Recrystallized
25	1.30	5.9	13.2	43	1 hour at 3000F
30	12.50	1.0	2.3	56	Material Reduced
25	7.09	2.0	4.3	58	65% - Stress
20	0.98	8.2	25.7	94	Relieved 1 hour at 2000F

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PRODUCERS OF TANTALUM AND TANTALUM PRODUCTS IN WESTERN EUROPE

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Imperial Metal Industries (Kynoch) Limited, P. O. Box 216,
Kynoch Works, Witton, Birmingham 6.
(Dr. N. P. Inglis and Mr. K. H. Wollaston)

Marston Excelsior Limited, Fordhouses, Wolverhampton.

Accles & Pollock, Oldbury, Birmingham.
(C.J.S. Cashmore)

GERMANY

Siemens und Halski, Siemensstadt, Berlin.

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(Dr. Hans-Joachim Friedrich)

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SECTION II

A STATE-OF-THE-ART-SURVEY OF FOREIGN TECHNOLOGY IN THE AREA OF TANTALUM ALLOY TUBING

INTRODUCTION

Three basic facts dominate developments in tantalum metallurgy: (1) Good tantalum ore is rare and the primary winning of tantalum is complex and expensive without immediate outlook of drastic cost reductions, (2) Although a refractory metal, tantalum belongs to a group of metals which oxidize readily at about 1000C setting a limit to high temperature applications. There is no prospect visible in Europe of overcoming this feature by alloying or treatment, (3) The consolidation and working of tantalum must proceed in vacuo or a protective atmosphere. Once this requirement is met, the working of tantalum, in the hands of experts, is considered to present few basic problems. European industry's response is "give us the order and we shall do the job."

Practically all tantalum extracted in Europe originates from columbite ore which is largely a mixture of the oxides of columbium and tantalum. Much of the ore is imported from East Africa. Other sources are the by-product of tin mining. The best ores for tantalum extraction have a 50/50 composition and the limit of economic extraction lies at 70/30 niobium/tantalum. Good quality ores are rare and expensive. Although the use of columbium as an alloying (stabilizing) element in stainless steels is rising, the use of columbium rich alloys is falling in Europe. Columbium in stainless steel is added mostly in the form of ferro-alloys and not as pure columbium won from columbium and tantalum ores. Hence, the columbium yield, an inevitable by-product in the winning of tantalum, does not reduce the price of tantalum.

The production of tantalum powder after extraction of the oxides, separation of the tantalum compound, and its purification is mainly carried out by a fusion electrolysis process originally developed at the U. S. Bureau of Mines. On an industrial scale, several American and European companies have developed independent techniques and know-how. Currently, the price of tantalum powder is of the order of $51.00 per pound. The total extraction in Europe is about 20 tons per annum, but a considerable expansion is imminent.

Although Siemens & Halske in Berlin were extracting tantalum powder before 1939, the earliest and still the largest powder plant in Europe using fusion electrolysis was started in 1945 and is still in operation by Murex. Murex have had a long history in the extraction and powder metallurgy of tungsten and molybdenum and were, therefore, well fitted to develop the industrial process. They claim that in the early stages they overtook the United States development by the Fansteel Metallurgical Corporation, the largest of the United States producers, but in recent years they believe that, in cost and quality, both producers are level, although the American production is considerably larger, about 150 tons per annum.

Other European extractors are Siemens & Halske, with a small output; Starck, Pechiney, with a small production; and Billiton in Holland (Billiton are associated with the American Kawecki Corporation) with a moderate production.

Two further companies are about to enter the field of tantalum extraction and re-processing. Hoboken have taken a license from the Fansteel Corporation and are said to be ready to start an extraction plant within a year. CIBA, Switzerland, have spent a great deal of money (reputedly 5-6 million dollars) to develop their own extraction process. The plant is now under construction. Its capacity is about 50 tons per annum of tantalum powder.

There is uneasiness in European industry about this sudden growth of potential. It is believed that the extensive development cost invested by CIBA was in part due to the lack of experience of that company in the extraction of metals. It has not resulted in either a cheaper or better process but was due to the unwillingness of CIBA to pay the price of a license.

INGOT PRODUCTION

Until recently, ingots were obtained from powder mainly by the classical powder metallurgy methods but with the sintering operation taking place in vacuo. The largest ingots obtainable, which are produced by these methods, for example at Plansee, weigh about 441 pounds. Similar limitations apply to other European producers of ingots. These include Murex, I.M.I., Billiton, Pechiney and Heraeus, and will shortly also include Hoboken.

It is stated that Pechiney are limited in the size of their ingots to four-inch diameter. Plansee have done some development on the production of hollow ingots by compacting powder, working and sintering. Heraeus state that densities of 14.0-15.0 are obtainable after compacting and sintering. One hundred percent densification reaching a density of 16.5 is obtainable by working and re-sintering.

There is no evidence that developments extending the powder metallurgical process (as pursued by Battelle in America) are taking place in Europe.

Plansee claim, however, that their sintered ingots are significantly better in purity than United States sintered ingots. In particular, the Plansee quality is adequate for nuclear fuel elements (one of the main Plansee tantalum products).

Recently, several of the ingot producers have changed over to electron beam smelting. According to some experts, a better quality is obtained and, if the plant can be properly utilized, appreciable cost savings are possible.

Murex have a plant capable of smelting up to five-inch diameter ingots of up to 1 m length. An electron beam plant, which may cost about $140,000 by itself and about $280,000 with all accessories and installation and may take something like $28,000 to run-in, requires some experience to operate for tantalum re-melting. There are, however, no fundamental difficulties and tantalum is considered one of the easiest metals to handle in this way.

I.M.I. are about to start an electron beam plant. Pechiney, Metaux Precieux are using both sintering and electron beam methods but Plansee are continuing with the sintering process exclusively. Even Heraeus, who are pioneers in electron beam gun development, are not yet certain whether they can abandon powder metallurgical techniques altogether.

The general advantages of the electron beam melting process are well known. Apart from maximum purification, these include an improved control of operational variables, more efficient utilization of available power and a wider choice of melting stock. The latter includes the re-cycling of scrap, of particular significance due to the high cost of tantalum.

Specifically, beam melted tantalum has an excellent surface. Heraeus state that only 0.5 mm had to be machined off ingots exceeding 50 mm in diameter. A typical purification achieved by beam melting is the reduction of oxygen (all figures in parts per million) from 83 to 6, of hydrogen from 115 to 1, of carbon from 14 to 18, of aluminum from 220 to 50 and of nitrogen from 35 to 10.

Among electron beam plants, the Heraeus gun method of beam melting is claimed to have several advantages over the ring method, namely longer cathode lives, more easily replaced cathods, less electron emission and no glare problems. Owing to good gas evolution associated with the large liquid surface open to vacuum at the top of the crucible, efficient de-gassing and purification are achieved. The success of Heraeus in selling plants outside Germany (e.g., I.M.I.) appears to confirm the competitive advantages of their plant.

I.M.I. purchase electron beam melted ingots from the Wah Chang Corporation, Albany, Oregon, until their own electron beam plant is in full operation, which is a 260 Kw four-gun unit (with provisions for other guns, if needed). This is a Heraeus plant capable of a maximum diameter of four inches and a maximum length of six feet.

I.M.I. feel that, to produce the best electron beam melted ingots, two melts would be needed. The first rapid melt for de-gassing and a second slow melt for final purification and surface quality.

Although I.M.I. believe that they can produce even larger tantalum ingots in their arc melting plant, no arc melted ingots are actually produced in Europe and no further developments of this process in the direction of a simpler and cheaper melting practice than the electron beam process, such as are proceeding in the United States,are envisaged in Europe.

Accles and Pollock report that electron beam melted material used by them has a higher ductility than sintered material (97% compared with 70%). In other respects their work is inconclusive on this point.

Heraeus use both sintered and electron beam melted ingots alongside each other. Further experience is required to standardize on one process. Russian metallurgists claim that the composition of tantalum powder can be prescribed to ensure almost complete C.O. and Ss purification after sintering.

TUBE MANUFACTURE

Tantalum tubes are produced in Europe by several methods. Deep drawing from sheet or foil and mandrel drawing, extrusion and drawing, machining from solid rod, welding up from sheet or foil and roll forming and welding from strip. Tubes are manufactured by Murex, I.M.I., Accles and Pollock in Great Britain, by Pechiney in France, by Heraeus in Germany and Plansee in Austria, and by Metaux Precieux in Switzerland.

Accles and Pollock, Heraeus, Plansee, Metaux Precieux, and others reduce tube by deep drawing from sheet. Accles and Pollock give the following details:

The process consists of making a cup and deep drawing down to size suitable for mandrel work. Cups are annealed (in vacuo) during deep drawing to prevent edge cracking. Usually, annealing takes place after 50-70 percent reduction.

Deep drawing is carried out using hardened and tempered, chromium plated punches with aluminium bronze dies. Punches have a taper of 0.001 inch per inch to prevent galling on extraction. Lubrication is by beeswax on an anodized film.

Mandrel drawing employs a steel mandrel (hardened and tempered) and the percentage of reduction depends on the original gauge. Accles and Pollock obtain 35

percent reduction in area per pass and up to 70 percent total reduction before annealing with heavy gauge (about 0.15 in.). More frequent annealing and lighter passes are needed for light gauge tubes to prevent splitting. The lubricant is graphite on an anodized film. Mandrels are withdrawn by reeling on wooden or fibre rolls (to prevent metal pickup off rolls).

Lubricant and anodized film are cleared off before vacuum anneal (10-4mm Hg). Annealing takes place at 1200C for 15 minutes (carefully controlled to prevent critical grain growth at base of cup. Anode film is pickled off in 5 percent HF- 40 percent HNO_3 solution). Dies used for mandrel drawing are of tungsten carbide.

Drawing speed is about 30 feet per minute.

It is stated that plug rolling is not yet possible, due to a tendency of tantalum to gall.

The starting material for mandrel is a sintered disc obtained from Murex (maximum size: 8 inches diameter and 0.15 inch thick).

Material sintered in tubular form has been found difficult to draw. It is felt, however, that this process has possibilities if further developed. The particular advantage is a possible reduction in the price of starting stock.

Production capacity is limited by demand which so far has not been much. Tube sizes are limited by size of starting stock (maximum tube size is 3/4 inch diameter at about .011 inch wall) and by annealing plant (limits maximum length to 40 inches). The smallest tube so far produced for a United States customer has a diameter of 1/32 inch with .002 to .003 inch wall, which was about 12 feet long (sent out unannealed). Accles and Pollock believe that .010 inch diameter (.002 inch wall) tube could easily be produced. All these tubes are made with tapered steel mandrels.

Present customers are mainly the U.K. Atomic Energy Authority, who have had tubes for research work, and Mullard Valves, who use small diameter tubes for growing silicon whiskers. Not much work done other than this. Output last year was only a few pounds.

Accles and Pollock prefer to develop the small bore tube business, particularly as the annealing can be done in coil form without long vacuum chambers.

I.M.I. have developed a cold extrusion process for tubes. The ingot is either pierced or bored to yield a bottle and then extruded in a 200-ton press by the inverted process, using a copper follower to ensure that all tantalum leaves the press and thus to increase the yield. It is believed that the lubrication in this process includes the use of a resin which has been developed for similar purposes and constitutes a trade secret.

I.M.I. also have a new tube drawing process. A collapsible, disposable copper mandrel is inserted and the rod so produced is drawn down. The mandrel is then removed either by cutting the tube into the required lengths and then pickling or by annealing the copper (in vacuo) and manually stripping it from the tantalum.

Another process developed by I.M.I. is the roll forming and welding from tantalum strip. Tubes either welded from sheet and/or roll-formed from strip and welded are sometimes drawn after welding. The minimum wall thickness before drawing is 0.020 inches. The welding is carried out as a separate operation with tungsten

electrodes in an argon filled chamber. Although identical in principle, the method is considered superior to the argon arc method performed in the open. I.M.I. possess a long argon chamber with a working space of 15 feet.

Experiments are proceeding with a rotary swaging process designed to reduce the wall thickness, while retaining the same diameter. Tubes have been made by this process, 1-inch in diameter and 0.010 inch in wall thickness.

Extruded and drawn tube is offered in a range of diameters between 0.1 and 0.75 inches. Wall thicknesses at the lower end of diameters can be as low as 0.005 inches and at the upper end of diameters 0.050 inches. The length of extruded tube is controlled by the maximum weight of the slug in the available equipment, which actually diminishes with diameter. At the larger diameters, the weight of the slug does not exceed two pounds.

The total output of I.M.I. is on a pilot scale and amounts to one ton per year. Process development is still proceeding and the company is anxious to improve its facilities and know-how.

There are some problems in tantalum processes. The metal tends to gall during extrusion and drawing. Annealing in a vacuum furnace is essential and sets the limits on sizes of individual pieces. The present vacuum furnace is only three feet long but is capable of annealing at 1750C. Larger furnaces are in use but with a limiting temperature of 950C.

I.M.I. have also carried out some work with 90Ta-10W alloy. The problems are those of proper melting of the tungsten component. This was satisfactorily solved by re-melting in the electron beam plant.

Metaux Precieux are partly newcomers and use drawing and forming from sheet for tube manufacture. The range of drawn tubes is from 3 to 30 mm diameter. Welded tubes up to 300 mm diameter (wall thickness 0.5 - 3 mm) have been made up to a length of 5 m. Metaux have indicated that they can carry out zone annealing on long tubes.

Typical ranges of sizes are given by Plansee as follows: Deep drawn tubes are offered with a bore from 3 to 20 mm, with a typical wall thickness of 0.2 - 0.3 mm. Tubes bored from solid start at 15 mm bore and go up to 75 mm. Typical wall thicknesses are between 0.2 and 3 mm. A tube of 25 mm bore may have a length of up to 1 m. Welded tubes are produced by fusion welding of the seam under an inert gas. The so-called Argon arc or Heliarc processes have proved completely satisfactory. Sheet is produced in widths of up to about 1 m.

Murex state the following limitations for seamless tube drawn from sheet: 1/16 inch - 1 inch diameter, 0.004 inch - 0.080 inch wall thickness. Tubes above 1-inch diameter can be fabricated from sheet by argon arc welding or resistance seam welding. The minimum wall thicknesses are: 0.025 inch for argon arc welded and 0.005 inch for seam welded tubes.

Heraeus state their range of tubes as follows: Seamless drawn tubes of up to 8 mm outer diameter are available in full millimetre graduations. Tubes from 71 mm up to 200 mm diameter are available with longitudinal welded seam. The average wall thickness is 0.3 to 0.4 mm. It is claimed that, by a special Heraeus process, very long tubes can be made. They can be supplied with one end closed and flanged. The total weight of a drawn tube is limited to 8 kg. Heraeus state that the gas content in tantalum is one of the few problems in

tube production. Professor Gebhart of the Max Plank Institute in Stuttgart is the German expert in this field.

There is no suggestion within the industry that the size limitations listed are in any way inherent to the material or the processes employed. Any substantial demand for tubes outside these limits could, within reason, be readily met by appropriate equipment and tooling.

Original Russian work is known on roll-forming and argon arc welding of tubes and subsequent rolling of the tubes (with three rollers over a short mandrel). Welded tubes were made in this manner of 6 - 75 mm diameter and 1 - 2.5 mm wall thickness. Rolling of annealed welded tube yielded thin-walled tubes of 6 - 30 mm diameter, 0.1 - 1 mm wall thickness and up to 8 m lengths.

No extensive applications exist for tantalum rich alloys. Few alloys are offered by European industry and the volume is small. Murex offer an alloy consisting of 90 percent tantalum and 10 percent tungsten which has a combination of higher strength but lower corrosion resistance than pure tantalum. Heraeus offer the same alloy. Tubes do not appear to have been made in this alloy.

Other tantalum rich alloys mentioned, partly in connection with nuclear work, contain 10 percent hafnium and 5 percent tungsten, or 30 percent columbium and 7.5 percent vanadium.

TUBE ASSEMBLIES

All the producers of tantalum tube in Europe also produce other tantalum semi-finished products such as rod, wire, sheet, foil and expanded mesh.

Murex, Heraeus, Plansee, Marston Excelsior, Pechiney and Metaux Precieux are also in the business of fabricating tantalum units for the chemical industry. These include manipulated tubes, tubes with flanges, corrugated tubes, machined components and complete assemblies.

Marston Excelsior process in a long argon chamber that can accommodate 15 feet long work pieces.

Apart from the steadily, though moderately expanding use of tantalum in capacitors, the outlets for tantalum products have been static in recent years. Nuclear reactor applications were considered and some experiments have been carried out but abandoned in Great Britain. No application is known or envisaged in European space technology and it is feared that the high rate of oxidation will render this and other high temperature applications difficult.

Most of the outlets outside the capacitor fields are in the chemical and allied industries. These outlets are unlikely to grow dramatically. Apart from the high cost (about 60 times the cost of stainless steel) the main reason is the temperature limitation for the corrosion resistance of tantalum. Although the need for more chemical processes conducted at high temperatures is increasing, this need cannot be fulfilled by tantalum. In the low temperature range, the volume expansion of the chemical industry is accompanied by steady improvement of materials such as glass and others as lining materials for tanks and pipework, so that tantalum apparatus remains reserved for the exceptional cases.

Heraeus feel that further demand for tantalum would increase rather than reduce the cost in the way that happened in some precious metals, e.g., platinum..

Beyond the over-all figures for European tantalum capacity, given earlier, which refer to production of tantalum powder (and therefore cover tantalum foil for capacitors as well as other products), year by year and country by country detailed statistics fluctuate greatly and are of little significance. A single important order in any given period entirely changes the position. Tantalum products are manufactured to customers' demands and nowhere in Europe is tantalum capacity fully utilized.

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SECTION III

PROPOSED PROGRAM FOR PHASES II, III AND IV

INTRODUCTION

The over-all objective of Contract AF33(657)-11261 is the development of manufacturing methods for the production of tantalum alloy tubing with improved high strength and corrosion resistance. The measure of complete development is the production of tantalum tubing in the Ta-10W and the T-222 (Ta-9.6W-2.4Hf-0.01C) alloys to the following sizes:

Outside Diameter (inches)	Wall Thickness (inches)	Length (feet)
0.500	0.062	20
0.375	0.062	20
0.250	0.020	20

having target properties of:

2400F	Ta-10W	T-222
a UTS - psi minimum	38,000	--
yield - psi minimum	27,000	--

The technical plan contained herein details a comprehensive program to achieve the goal based on the current state-of-the-art.

An important consideration for an economical production process for tantalum alloy tubing is the availability of high quality tube hollows for subsequent reduction. It is proposed that the combination of extruding and tube reducing followed by a sequence of drawing operations, with intermediate vacuum stress-relieving or recrystallization anneals, be utilized in producing the desired tubing. The other major processes for producing tube hollows, tre-panning, piercing, etc., have a serious disadvantage because of their length limitations. We intend to extrude tube hollows in lengths in excess of ten feet. By this achievement, higher yields and obvious economic advantages can be realized and as a result a unique contribution can be made to the state-of-the-art.

The tube reducing method in commercial use on some 150 other ferrous and non-ferrous metals offers a practical method for obtaining a preliminary evaluation of the fabricability of each of the tantalum base alloys, at the same time permitting a reasonably large O.D. reduction in combination with excellent economy of materials. The amenability of the Ta-10W alloy to commercial tube drawing practices has been demonstrated. It is anticipated that the T-222 alloy will likewise be amenable to commercial drawing practices.

PHASE II - DEVELOPMENT OF TUBE HOLLOWS

This phase concerns the development of a manufacturing process for the production of quality tube hollows. The following proposed procedure is based on the best current knowledge of the state-of-the-art and best extrusion know-how at Allegheny Ludlum.

Materials

Reliable technology to produce sound and reproducible ingots of the Ta-10W alloy has been developed on Navy Special Projects Office Contract NOrd 18787, "Development of Tantalum-Tungsten Alloys for High Performance Propulsion System Components," and U. S. Air Force Contract AF33(600)-42396 "Tantalum Extrusion Program" T-222 is an experimental alloy developed on Navy Contract N600(19)-59762 "Pilot Production and Evaluation of Tantalum Alloy Sheet." The largest melt, to date, is three-inch diameter by 22 pounds. However, this alloy is a modification of the commercially available T-111 alloy and reliable producers of refractory metal alloys have quoted on supplying wrought, fully machined extrusion billets of the size required on this program. We intend to purchase fully machined, ready to extrude, hollow extrusion billets $4.850^{+0.015}_{-0.000}$ inch O.D. by $1.6^{-0.015}_{+0.000}$ I.D. by 11 inches long and having a surface finish of 62 RMS or better. In doing this we will obtain the highest scrap economy by having the major portion of billet scrap generated at the source, and minimize scrap control at our location. The material used on this program must conform to the following requirements:

1. The Ta-10W alloy will be prepared by either double electron beam melting or a duplex system consisting of a primary electron beam melt followed by consumable vacuum arc melting. The T-222 alloy will be prepared by double consumable vacuum arc melting.

2. Certified billet soundness in accordance with the ultrasonic procedure described in Ordnance Specification OS-9426-A. Acceptance level to be described as Quality Level I in Specification OS-9426-A.

3. Machined and ground surface finish equal to or better than 62RMS.

4. Free of injurious defects such as seams, pipe, cracks, scale, nonmetallic inclusions and segregation which adversely affect extrudability.

5. Billet ends checked by dye penetrant method.

6. Billet concentricity $\pm$ 5 percent.

7. Recrystallized microstructure with uniformly fine grain size equal to or smaller than ASTM No. 3.

8. Certified chemical analysis on turnings from the end face of one billet from each heat, with spot checks on other faces.

9. Chemical specifications are as follows:

Chemical Specifications

Ta-10W Element	Ta-10W Maximum Weight %	T-222 Element	T-222 Maximum Weight %
W	9-11	Chemical Specification Not Yet Available.	
O	0.010		
N	0.005		
C	0.005		
Cb	0.010		
Fe	0.005		
Co	0.002		
Mo	0.020		
Ni	0.005		
Si	0.015		
Ti	0.002		

Material Evaluation

In addition to a thorough inspection to establish billet soundness by non-destructive methods, an evaluation of all billet stock will be done to define metallurgical and mechanical properties. A disc will be obtained from each billet for examination of the microstructure, grain size and micro-porosity. Hardness surveys will be taken in order to indicate and measure the uniformity and effect of previous history and heat treatment.

Extrusion

As the result of the past two years of extrusion development in support of various Air Force programs, a unique tooling system for direct extrusion has been designed and made to perform beyond the normal limits of temperature and pressure for hot extrusion.

We propose to develop the extrusion process for this phase on a conventional Lake Erie production press of a modified Schloemann design. The press is rated at 2200 tons.

We propose to produce a 2-inch O.D. by 0.250-inch wall tube hollow by extrusion from a 5-inch diameter liner. The full capacity of 2200 tons will be utilized to give, in effect, a liner pressure of 224,000 psi. Current extrusions of Ta-10W "T" shapes on Contract AF33(600)-42396 indicate the resistance to deformation is about 70,000 psi from a billet heating temperature of 3350F at a reduction ratio of about 20:1. For the extrusion of the tube hollow, a unit billet pressure of 246,000 psi will be in effect at the maximum liner capacity.

The extrusion of the 2-inch O.D. by 0.250-inch wall tube hollow from the 5-inch diameter liner contemplated is a reduction ratio of 13:1. The extrusion constant, K-value, as determined by the formula $P = K \ln R$ where P is the liner pressure in psi, K is the extrusion constant in psi, and R is the reduction ratio, would be 96,000 psi under these conditions. Since this value is considerably above the experienced K-value of 70,000 psi, it is believed that the tube hollows for the program can be readily achieved from a billet temperature of 3350F. Extrusion

lubricants and tooling technology will be essentially the same as was developed on Contract AF33(600)-42396. From this relatively high reduction ratio in association with this unique, high performance tooling system, it is anticipated that extrusion lengths in excess of ten feet are possible. Obvious economic advantages can be realized with this capability.

Two short billets of each alloy will be extruded to the 2-inch O.D. by 0.250-inch wall tubing using two different extrusion conditions. One billet of each alloy will be extruded from a temperature of 3350F and,depending upon the results, the second billet will be extruded from either a lower or higher temperature.

These tubes will be supplied to our subcontractor, Superior Tube Company, for the initial tube reducing and drawing experiments under Phase III. Based on their performance during the initial tube reducing operation, the optimum extrusion condition will be selected for each alloy and the remaining billets will be extruded to tube hollows utilizing these optimum procedures. The goal for wall variation shall be ± 5 percent.

Extrusion Tooling

We propose to use an extrusion billet size of 4.850-inch outside diameter by 1.600-inch inside diameter by 10.6 inches long. These billets will be extruded on our 2200-ton capacity press. A tooling arrangement for the press is shown in Figure 20.

1. Liner Design and Materials

The extrusion liner for this program will be 5.000-inch diameter in which 224,000 psi liner pressure can be applied. With this liner size, a tube hollow can be extruded with a 2-inch outside diameter by 1/4-inch wall thickness. This is an extrusion ratio of about 13:1.

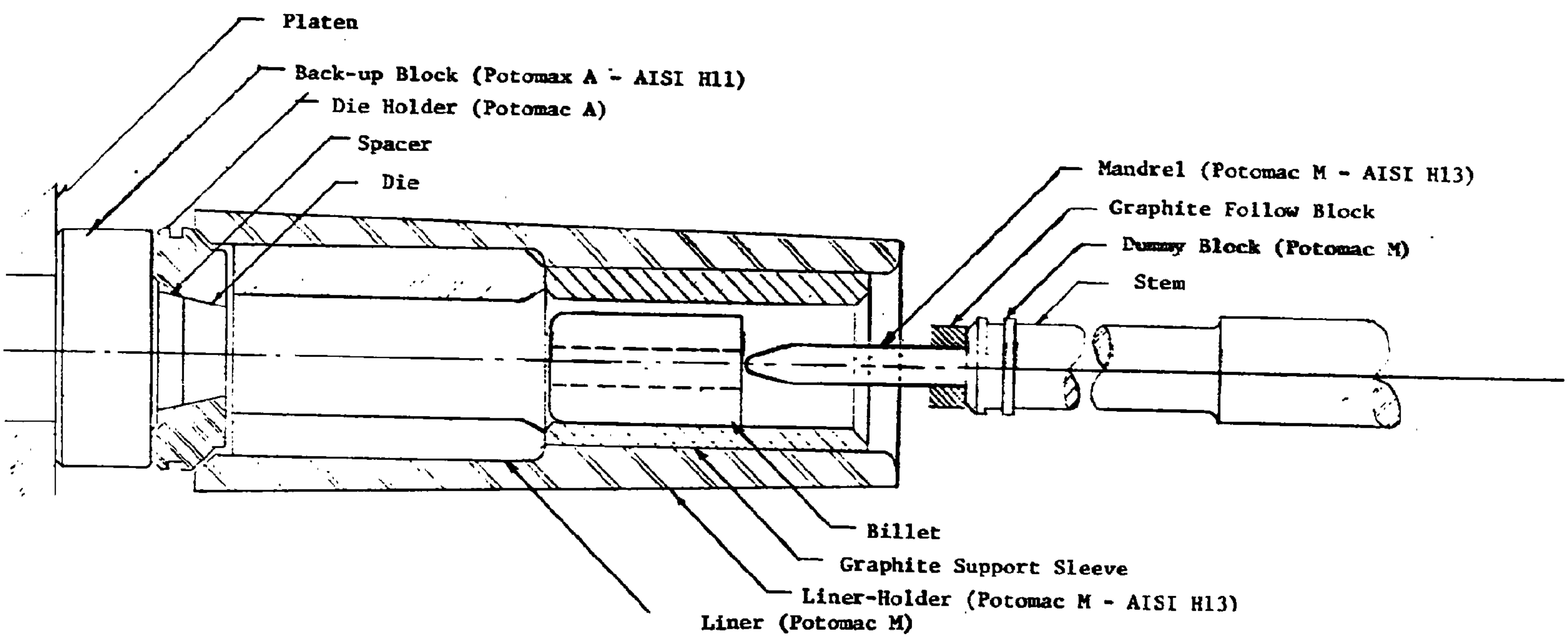
We propose to use a liner, liner-holder assembly for this program. The inner, working liner will be constructed of Potomac M AISI H13 alloy heat treated to Rockwell C 53-56. In order to reduce erosion and wear by the hot billet, the liner I.D. surface will be nitrided by the Nitrocycle process which is the only means of obtaining a nitrided case, free of brittle "white layer" and intergranular nitrides. This liner will be assembled in a liner-holder with an interference fit of 0.010 inch. The liner-holder will be made of AISI H13 alloy heat treated to Rockwell C 48.

The liner, liner-holder assembly will be placed in the container on a taper with an included angle of three degrees. The container is made of SAE 6145 steel heat treated to hardness level of Brinell 245-275.

The container temperature will be maintained at 500F primarily to avoid excessive loss of strength in the tooling components. It is interesting to note that excellent surface quality (50-95 RMS) has been achieved on unalloyed tungsten "T" shape extrusions produced by Allegheny Ludlum using this low container temperature.

2. Stem Design and Materials

We propose to use a special stem that can withstand an applied compressive stress up to 240,000 psi, which relates to a liner pressure of 224,000 psi. The stem will be constructed so that the high stress (240,000 psi) portion of the stem is confined within the liner-holder. A cast brass ring will ride on



Tooling Arrangement for the Extrusion of Tube Hollows

Figure 20

the bigger back end of the stem where the stress is below 200,000 psi. The cast brass ring seats against the container in order to prevent stem fragments from coming out as a result of an explosive type failure.

3. Die Design and Materials

We propose to use a die design for this program with a conical entry of 130 degree included angle, a constant entry radius of 5/16-inch and a constant land width of 1/4-inch. The die will be made of AISI H13 hot work tool steel heat treated to Rockwell C 48-50.

A thermal barrier of zirconia will be applied by the oxyacetylene flame spray process with solid rod feed. After coating, the die will be ground and polished with silicon carbide wheels and abrasive paper to 35-50 RMS surface finish.

4. Mandrel Design and Materials

We propose to use a mandrel design with a taper of 0.001-inch per inch of length and a conical nose configuration. Each mandrel will be made of AISI H13 hot work tool steel. A thermal barrier of zirconia will be applied by the oxyacetylene flame spray process with a solid rod feed. After coating, each mandrel will be ground and polished with silicon carbide wheels and abrasive paper to a 35-50 RMS surface finish.

Post-Extrusion Operation

1. Cleaning

Following extrusion, tube hollows will be cleaned by grit blasting with alumina, and visually inspected for defects.

2. Stress-Relieving

Stress-relieving of the tube hollows will be done in an argon atmosphere furnace.

3. Straightening

Following stress-relief, the extrusions will be straightened in a 100-ton capacity stretch-straightener with a 360-degree twisting head. If heating is necessary, a self-resistance heating unit is available adjacent to the straightener.

4. Conditioning

Facilities are available for honing the I.D. of the tube hollows in order to obtain a surface finish less than 125 RMS and free of contamination. Both centerless and spot grinding facilities are available for O.D. conditioning. Ends will be trimmed and samples obtained for laboratory evaluation.

5. Inspection

The following inspections and non-destructive tests will be conducted:

a) 100 percent dimensional check (vidigage)
b) 100 percent visual on O.D. for surface defects
c) 100 percent boroscope on I.D. for surface defects
d) 100 percent dye penetrant tests
e) 100 percent eddy current for surface defects
f) 100 percent ultrasonic for internal defects
g) 100 percent surface roughness determination

Testing (Tube Hollows)

End samples from each extrusion will be evaluated in terms of:

a) Hardness
b) Room temperature tensile tests
c) Metallographic examination
d) Analysis for interstitial elements-carbon, nitrogen, oxygen and hydrogen

Samples will be obtained to determine the stress-relieve and recrystallization temperature.

PHASE III - DEVELOPMENT OF TUBE PRODUCTION PROCESS (Test Lot Sequence)

The major work requirements under this phase will be subcontracted to Superior Tube Company of Norristown, Pennsylvania.

In view of the fact that commercial quality tubing has been produced from the Ta-10W alloy using commercial tubing facilities, it is believed that the primary objective of this investigation is to optimize the processing sequence required to produce finished tubing as related to the final quality standards and high temperature mechanical property characteristics. A second objective of this phase is to evaluate the acceptability of the finished tubing and to establish tentative specification requirements as to tolerances, quality and mechanical properties.

Criteria and Approach

It is expected, in view of the mechanical property goals at 2400F, that the optimum processing sequence must consider the over-all influence of the intermediate reductions as affected by the stress-relieving and recrystallization annealing treatment, in that the recrystallization temperature for each of these alloys may be above or below this testing temperature as related to their prior history. It is proposed, therefore, that the mechanical property requirements form at 2400F a control point in evaluating the acceptability of the sequences used for producing tantalum alloy tubing 0.500-inch O.D. by 0.062-inch wall, 0.375-inch O.D. by 0.062-inch wall, and 0.025-inch O.D. by 0.020-inch wall in 20-foot minimum random lengths.

The effect of prior thermal-mechanical history is considered of major significance in the ability of the tantalum alloys to sustain reliable strength levels above their recrystallization temperatures. For this reason, not only will the final and penultimate reductions be controlled, but the antipenultimate reductions as

well. If these reductions are significant, as past experience has indicated, then the thermal history must also be optimized. The effects of recrystallization as compared to stress-relieving will be examined.

Portions of an initial representative extrusion(s) 2-inch O.D. by 0.250-inch wall by 4-6 feet of each of the alloys will be divided into test lots in order to produce finished tubing of each of the designated sizes. For each size, several test lots will be required in order to develop an optimum thermal-mechanical history. The final cold reduction will be varied on the 0.250-inch O.D. by 0.020-inch wall size so as to provide hot tensile test samples in the 1/4 Hard, 1/2 Hard, and Full Hard tempers as well as the recrystallized and stress-relieved temper. An investigation of included die angles ranging from 24-80 degrees will be included. Tungsten carbide die material will be used. Drawing speeds in excess of 40 feet per minute will be utilized.

Upon completion of the hot tensile test results conducted on the test lot samples, the optimum process sequence will be selected and the material remaining from the initial representative extrusion processed for further evaluation. The goals for the dimensional tolerances shall be for the O.D. ± 0.002-inch and for the I.D. ± 0.0015-inch which is less than the half commercial tolerances requested in this proposal request.

Evaluation of the finished tubing in each of the three sizes of each tantalum alloy will include the following nondestructive testing:

a) 100 percent dimensional check (vidigage)
b) 100 percent hydrostatic test
c) 100 percent dye penetrant test
d) 100 percent eddy current test
e) 100 percent ultrasonic test
f) 100 percent helium leak test

In addition, samples from each of the sizes of finished tubing from each tantalum alloy shall be submitted to the following destructive tests:

a) Flaring, flattening and bending
b) Burst testing (0.250-inch O.D. by 0.020-inch wall only)
c) Tensile testing at room temperature (to establish hydrotest pressures to be used above)
d) Analysis for interstitial elements carbon, oxygen, hydrogen and nitrogen
e) Metallographic examination
f) Surface roughness
g) Weldability tests

Because of the suitability of existing proprietary lubrication techniques, further investigation of this specific factor is not planned. Proprietary lubrication procedures for reactive metals, including these tantalum alloys, have already been established by the subcontractor.

Upon completion of the above investigation and inspection results, the tentative specification requirements with a 20-foot minimum random length of tubing to be produced under Phase IV, will be established. It is estimated that the amount of tubing produced under Phase III, including that used for the various

tests, will be approximately 15 to 20 feet for the 0.500-inch O.D. by .062-inch wall size, as well as the 0.375-inch O.D. by 0.062-inch wall, and approximately 60 feet for the 0.250-inch O.D. by 0.020-inch wall size.

PHASE IV - PRODUCTION OF TUBING IN 20-FOOT LENGTHS

Objective

The objective of this phase is to produce finished tubing and to establish the reliability of the final product with respect to the tentative specification requirements developed under Phase III.

Criteria and Approach

From the balance of the extruded tubing remaining from Phase II at a size of 2-inch O.D. by 0.250-inch wall, the following tubing, aiming for 20-foot minimum random lengths, will be produced.

	Approx. Ratio by Weight (%)	90 Ta-10W Goal (ft.)	T-222 Goal (ft.)
0.500-inch O.D. by 0.062-inch wall	40	160	160
0.375-inch O.D. by 0.062-inch wall	40	240	240
0.250-inch O.D. by 0.020-inch wall	20	480	480

The above tubing to be tested for dimensional tolerances and for conformance to the tentative specification requirements developed under Phase III.

Acceptable finished tubing shall be identified and any material which does not conform to the tentative specification shall be so marked and identified.

Physical and mechanical property evaluation under Phases II, III and IV will be run in accordance to the following schedule:

TESTING SCHEDULE

Phase II - Development of Tube Hollows

	Tensile Room Temp.	Micro and Hardness Survey	Heat Treat Studies	Bend Tests
Ta-10W alloy 2 extrusions	8	4 sections	6 specimens	8
T-222 2 extrusions	8	4 sections	6 specimens	8

Phase III - Development of Tube Product Process

	Tensile				Creep Rupture		
	Room	2100F	2400F	2700F	2100F	2400F	2700F
Ta-10W Alloy							
1/2-inch O.D. Tube	9	9	9	9	9	9	9
3/8-inch O.D. Tube	9	9	9	9	9	9	9
1/4-inch O.D. Tube	9	9	9	9	9	9	9
T-222							
1/2-inch O.D. Tube	9	9	9	9	9	9	9
3/8-inch O.D. Tube	9	9	9	9	9	9	9
1/4-inch O.D. Tube	9	9	9	9	9	9	9

Phase IV - Production of Tubing

Ta-10W	Tensile 2400F	Creep Rupture 2400F
1/2-inch O.D. Tubes	4	4
3/8-inch O.D. Tubes	6	6
1/4-inch O.D. Tubes	8	8
T-222		
1/2-inch O.D. Tubes	6	6
3/8-inch O.D. Tubes	8	8
1/4-inch O.D. Tubes	16	16

The room temperature tensile tests, weldability studies, metallographic work, hardness surveys, heat treat studies and bend transition tests will be run at and by Allegheny Ludlum or its subcontractor, Superior Tube Company. All of the elevated temperature tensile and stress rupture testing will be done at outside sources.

APPENDIX A

COMPANY________________________ DATE________________________

Q U E S T I O N N A I R E

STATE-OF-THE-ART-SURVEY

Tantalum Alloy Tubing Development Program

Contract AF33(657)-11261

SECTION I

Interests, Applications, and Requirements for Tantalum Alloy Tubing

		Yes	No
A.	Do you have any specific needs for tantalum alloy tubing?		
	1. At present	___	___
	2. In the near future	___	___
	3. In the distant future (five years or more)	___	___

B. If a user or potential user of tantalum alloy tubing, please provide the following information:

1.

Type of Application	Alloy Composition	Tubing Size OD	Wall	Length	Supplier

2. Minimum Properties Required

	Temperature, °F 75	2000	2400	2700	3000	3500
Ultimate Strength						
Yield Strength						
% Elongation						
% Reduction in Area						
Minimum Bend Radius						
Creep Strength						
Rupture Strength						
Service Environment						
Hardness						

3. Other properties or characteristics required ____________________

____

4. Comments on presently available tantalum alloy tubing ____________________

____

C. If a producer of tantalum alloy tubing please supply the following information pertaining to your product:

Alloy Composition	Tubing Sizes Available
____________	____________
____________	____________
____________	____________

<u>Note</u>: If available, will you please attach any literature or brochures describing these products.

COMPANY____________________ DATE____________________

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SECTION II

Raw Materials

1. Are you a supplier of tantalum powder?____________________

2. Please list your available types and grades of tantalum powder

Grade Designation	Starting Material	% Purity (incl. Gases)	Average Particle Size

3. Please specify impurity levels in the tantalum powders used for alloying

Impurity	Maximum %
C	
N	
O	
H	
Cb	
Fe	
Ni	
Si	
Ti	
W	
Others	

4. Please specifiy impurity levels in the alloying powders used________________

____

____

5. Please supply pricing and availability information on the tantalum powders and alloying powders produced by your company________________________________

____

____

COMPANY________________________ DATE________________________

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SECTION III

Consolidation of Raw Materials

1. Do you supply pressed and sintered tantalum alloy electrodes for arc-casting? ____________ Pressed and sintered tantalum alloy billets for direct forging? ____________ Pressed and sintered tantalum alloy billets for direct extrusion? ____________________________

2. What tantalum alloy compositions do you supply in pressed and sintered ingots?

   ____

3. Please indicate the form of the raw materials (particle size, etc.) used for compacting pressed and sintered billets for use as:

   a. Arc casting electrodes ____________

   b. Direct forging billets ____________

   c. Direct extrusion billets ____________

4. How are the powders blended and what are the distribution characteristics of the alloy and tantalum powders? ____________________________

   ____

5. What general compacting procedures (mechanical pressing, hydrostatic pressing, etc.) are used? ____________________________

   ____

6. Compacting details:

Alloy	Method	Pressure	Billet Shape	Dimensions of Largest Shape Produced	As Pressed Density
______	______	______	______	______	______
______	______	______	______	______	______
______	______	______	______	______	______
______	______	______	______	______	______

7. Please give details such as type of heating, maximum temperature, billet size capability and atmospheres for the sintering furnaces available at your installation____

____

8. Sintering details:

Alloy	Maximum Billet Size	Time	Temperature	Atmosphere	Density	
					Initial	Final
______	______	______	______	______	______	______
______	______	______	______	______	______	______
______	______	______	______	______	______	______

9. Have you encountered any reactions with furnace hearth materials during sintering?______________If so, please give details______________________

____

____

10. Are claddings or coatings used to prevent contamination during sintering? ______________If so, please give details______________________

____

____

11. From your experience, what are the approximate diffusion rates of the alloy additions in tantalum______________________________________

____

____

12. What dimensional tolerances can you hold on pressed and sintered billets

Cross Sectional______________________________

Length______________________________________

Straightness_________________________________

13. What inspection methods are used to insure soundness and homogeneity of pressed and sintered billets

Penetrant Methods

Ultrasonic

Magnetic Susceptibility

Magnetic Particles

Other

COMPANY________________________________DATE________________________________

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STATE-OF-THE-ART-SURVEY

Tantalum Alloy Tubing Development Program

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SECTION IV

Melting Techniques

A. General

1. Are you a supplier of tantalum alloy melted ingots?____________________

2. If a supplier please list below the maximum ingot sizes available in the various alloys

Composition	Ingot Diameter	Weight
________	________	____
________	________	____

3. What general melting methods are employed at your facility?

Consumable Electrode______________

Electron Beam____________________

Other_________________________

4. What impurity levels do you specify for tantalum melting stock?

C

N

O

H

Cb

Fe

Ni

Si

Ti

W

Other

5. Is tantalum scrap utilized in your melting practice?____________If so, to what extent?______________________________

6. Is multiple melting utilized to insure ingot homogeneity?______________

   Please specify______________________________

B. Consumable Electrode Melting

1. What method is employed to join electrode sections?

   Threaded Nipple__________________

   Welding__________________

   Other__________________

   (If welding, specify practice used)

2. What electrode configurations are used?______________________________

   ____

3. What crucible construction is used?______________Material____________

   Wall Thickness____________________Taper____________________

   Liners (if used)______________________________

4. What is the size of the electrode in relation to the size of the crucible?

   ____

5. According to your experience, what are the optimum melting conditions for the various tantalum alloys?

Alloy Composition	Electrode Diameter	Ingot Dia.	Voltage	Amps	Polarity	AC or DC	Furnace Atmosphere	Melt Rate (lbs/min)

6. Is stirring used during melting?____________If so, please specify__________

   ____

7. Is hot topping practiced?________________If so, please specify procedures?______________________________

8. Are deoxidizing agents used?________________If so, please specify________

____

9. Are grain refining additions used?______________If so, please specify_____

____

10. What is your normal yield from electrode to conditioned ingot?____________

C. Electron Beam Melting

1. According to your experience, what are the optimum melting conditions for the various tantalum alloys?

Alloy Composition	Ingot Dia.(in)	Ingot Length(in.)	Power Required	Vacuum Pressure	Melting Rate(lbs/min)	Average Hardness

2. Are grain refining additives used?__________If so, please specify__________

____

3. Are deoxidizing additions used?_____________If so, please specify________

____

4. What is your normal yield from electrode to conditioned ingot?____________

COMPANY________________________ DATE________________________

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SECTION V

Ingot or Pressed and Sintered Billet Evaluation

A. Inspection for Homogeneity

1. What inspection methods are used to insure ingot soundness?________________

   ____

   Please indicate effectiveness of each method________________________

   ____

2. What surface conditions are normally obtained on arc-melted ingots and pressed and sintered billets?________________________

   ____

3. If available, please submit photographs of typical macro and microstructures obtained on the as-cast ingots and the pressed and sintered billets________

   ____

4. Have homogenizing heat treatments been effectively used to obtain a more homogeneous structure?____________________Please specify the effect and the treatment used for the various tantalum alloys________________

   ____

   ____

5. Have consumable arc, electron beam, and powder metallurgy techniques been compared on one tantalum alloy? If so, what alloys?________________

   ____________________What method or combination of methods proved best?

   ____

   ____

B. <u>Ingot Conditioning</u>

1. What method of cropping is normally used in conditioning ingots?__________

   ____

2. What method of surface conditioning is most effective?__________________

   ____________________What is the finest surface obtainable by this

   method?____

3. Ingot conditioning losses:

   Average shrinkage,%_______________Surface conditioning plus clean-up

   loss,%_________________________Hot top loss, %__________________

   Butt loss,%________________________Average over-all recovery__________

   ____

COMPANY________________________________ DATE____________________________________

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SECTION VI

Initial Ingot Breakdown

If you are a fabricator of tantalum alloys please complete this section.

A. General

1. What method is used for initial ingot breakdown of both pressed and sintered and arc-melted tantalum alloys?

Alloy	Pressed and Sintered	Arc-Melted Consumable	Arc-Melted Electron Beam

2. Please describe any laboratory-scale tests you have performed which correlate with the hot-workability of tantalum alloys____________________

____

B. If Forging or Rolling is Used for Initial Ingot Breakdown, Please Complete the Following:

Alloy Composition	Working Operation	Billet Size	Pre-heating Temp(°F)	Type of Heating	Time to Temp.	Lubricant or Protective Medium Used

% Reduction per pass	Reduction Between Anneals	Annealing Conditions		
		Temp.	Time	Atmosphere

C. If Extrusion is Used in Breakdown, Please Complete the Following:

Alloy Composition	Billet Dia.	Reduction Ratio	Extrusion Temp°F	Method of Heating

Time to Temp.	Heating Rate	Heating Atmosphere	Lubricant or Cladding	Maximum Extrusion Pressure

COMPANY_______________________________DATE_______________________________________

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SECTION VII

Post Extrusion Operation

If You Are an Extruder of Tantalum Alloys, Please Complete the Following Section.

A. Surface Conditioning

1. Please describe pickling techniques used for the various tantalum alloys.

Alloy	Pickling Solution	Time	Solution Temp.
____	____________	____	__________
____	____________	____	__________
____	____________	____	__________

2. Please describe any mechanical conditioning performed on extruded products.

____

____

3. Please describe the inspection techniques used for determing quality of the extrusion.____

B. Straightening

1. Please describe the method and equipment used to straighten the extrusions___

____

____

Is straightening done warm or cold?______________________________________

____

2. What conditioning is necessary following the straightening operation?________

____

____

3. Please describe the inspection techniques used to determine straightness of the finished extrusion________________________

C. Detwisting

1. Please describe the equipment and techniques, including temperatures used to detwist the extrusions________________________

2. What conditioning, if any, is required following the detwisting operation?________________________

3. What inspection techniques are used to determine degree of twist in final product?________________________

D. Heat Treatment

		Stress Relief		Recrystallization			
Alloy	Temp. °F	Time	Atmosphere	Temp. °F	Time	Atmosphere	% Recrystallization

E. Drawing Information

	1	2	3	4
Alloy				
Piece (round) (hollow) (shape)				
Type of Draw (plug) (mandrel) (sink)				
Bench Type				
Bench Capacity				
Die Type				
Speed (ft/min)				
Stock Temp (°F)				
Tooling Temp (°F)				
Lubricant				
% Reduction/pass				
No. of passes between anneals				

F. Please describe any other post-extrusion operations, including tube reducing, swaging, rolling, forging, etc., utilized to produce a finished product__________

____

____

____

NOTE: If methods other than extrusion are used to produce tube hollows, please specify and give details of the operation____________________

____

____

____

G. Mechanical Property Evaluation

Please complete the attached alloy data sheets for each of the alloys you have produced or have used and tested.

COMPANY________________________________ DATE________________________________

TANTALUM ALLOY DATA SHEET

Alloy Composition________________________ Melting Point____________________

Consolidation and Fabrication History____

____

____

Condition (as-extruded, stress relieved, recrystallized, % cold worked, etc.)________

____

____

Heat Treatment____

____

TENSILE DATA

Test Temp °F	Strain Rate____ 0.2% Y.S. (psi)	T.S. (psi)	%EL	%RA	Strain Rate____ 0.2% Y.S. (psi)	T.S. (psi)	%EL	%RA

CREEP DATA

	Stress To Produce % Strain								
	Hours			Hours			Hours		
Temp °F	%	%	%	%	%	%	%	%	%

COMPANY_______________________________ DATE_______________________________

STRESS RUPTURE DATA

Temp (°F)	Atmosphere	Stress (psi)	Life (hrs)	%EL	%RA	Comments

HARDNESS

Room Temp	°F	°F	°F	°F	°F

OXIDATION DATA

Temp(°F)	Time	Atmosphere	Weight Gain	Penetration (mils/side)	Metal Loss (mils/side)

IMPACT DATA

	RT	°F	°F	°F	°F
Charpy					
Izod					

BEND TRANSITION

Temp________°F Speed________in/min Angle________°

COMPANY______________________________ DATE______________________________

METALLOGRAPHIC

ASTM GRAIN SIZE____________ INCLUSIONS____________

NOTE: Please list pertinent references and attach data which supplements the information requested above______________________________

COMPANY______________________________DATE______________________________

TANTALUM ALLOY DATA SHEET

Alloy Composition__________________________Melting Point__________________________

Consolidation and Fabrication History____

____

____

Condition (as-extruded, stress relieved, recrystallized, % cold worked, etc.)_________

____

____

Heat Treatment____

____

TENSILE DATA

Test	Strain Rate____				Strain Rate____			
Temp °F	0.2% Y.S.(psi)	T.S.(psi)	%EL	%RA	0.2% Y.S.(psi)	T.S.(psi)	%EL	%RA

CREEP DATA

	Stress To Produce % Strain								
	____ Hours			____ Hours			____ Hours		
Temp °F	___%	___%	___%	___%	___%	___%	___%	___%	___%

COMPANY________________________ DATE________________________

STRESS RUPTURE DATA

Temp (°F)	Atmosphere	Stress (psi)	Life (hrs)	%EL	%RA	Comments

HARDNESS

Room Temp	___°F	___°F	___°F	___°F	___°F

OXIDATION DATA

Temp(°F)	Time	Atmosphere	Weight Gain	Penetration (mils/side)	Metal Loss (mils/side)

IMPACT DATA

	RT	___°F	___°F	___°F	___°F
Charpy					
Izod					

BEND TRANSITION

Temp________°F Speed________ in/min Angle________°

COMPANY________________________DATE________________________

METALLOGRAPHIC

ASTM GRAIN SIZE__________INCLUSIONS__________

NOTE: Please list pertinent references and attach data which supplements the information requested above

APPENDIX B

QUESTIONNAIRE DISTRIBUTION LIST

Aerojet General Corporation
P. O. Box 296
Azusa, California
Attnetion Mr. L. Gilbert, Head
Materials and Processes

Argonne National Laboratory
Lemont, Illinois
Attention Mr. James Schumar

Armour Research Foundation
Metals Research Department
Technology Center
Chicago 16, Illinois
Attention Dr. William Rostoker
Assistant Manager

Atlantic Research Corporation
Alexandria, Virginia
Attention Mr. E. Olcott

AFSC Aeronautical Systems Division
Manufacturing and Materials Technology
Wright-Patterson Air Force Base,
Ohio
Attention - LMBMB
Major C. M. Hollyfield

Babcock and Wilcox Company
Beaver Falls, Pennsylvania
Attention Mr. John B. Rutherford
Chief Metallurgist

Battelle Memorial Institute
505 King Avenue
Columbus 1, Ohio
Attention Mr. D. Maykuth

Beech Aircraft Corporation
Wichita, Kansas
Attention Mr. C. A. Rembleske
Chief Administrative Engineer

Bell Aerosystems Company
P. O. Box 1
Buffalo 5, New York
Attention Mr. George Kappelt, Director
Engineering Aero-Space Rockets

Bendix Aviation Corporation
Bendix Products Division
401 N. Bendix Drive
South Bend 20, Indiana
Attention Mr. W. O. Robbinson

J. Bishop and Company
Malvern,
Pennsylvania

Boeing Airplane Company
Wichita 1, Kansas
Attention Mr. C. B. Barlow
Chief Design Services

Boeing Airplane Company
P. O. Box 3707
Seattle 24, Washington
Attention Mr. J. T. Stacy

Bridgeport Brass Company
Bridgeport 2, Connecticut
Attention Mr. R. S. French
Chief Metallurgist

Bureau of Mines
Albany, Oregon
Attention Mr. A. H. Robinson

Bureau of Mines
P. O. Box 136
Rolla, Missouri
Attention Mr. J. A. Rowland
Research Director

California Institute of Technology
Jet Propulsion Laboratory
Pasadena, California
Attention Mr. Donald P. Kohorst

Calumet & Hecla, Inc.
Wolverine Tube Division
1411 Central Avenue
Detroit 9, Michigan
Attention Mr. G. L. Craig, Director
Research and Development

Canton Drop Forging and Manufacturing Company
17th Street Extension S. W.
Canton, Ohio
Attention Mr. Frank J. Welchner
Metallurgist
Quality Control Manager

Chance Vought Aircraft, Inc.
P. O. Box 5907
Dallas, Texas
Attention Mr. Milton J. Rudick
Chief of Applied R & D
Astronautics Division

Chase Brass and Copper Company
Waterbury, Connecticut
Attention Dr. D. K. Crampton
Director of Research and
Development

Chromalloy Corporation
169 Western Highway
West Nyack, New York
Attention Dr. R. A. Cooley

Climax Molybdenum Company of
Michigan
14410 Woodrow Wilson Boulevard
Detroit 3, Michigan
Attention Mr. George A. Timmons
Director of Research

Comptoir Industriel d'Etirage
et Profilage de Metaux
30 Avenue de Missine
Paris 8e
France
Attention Monsieur J. Sejournet
General Manager

Convair
Division of General Dynamics
Corporation
San Diego 12, California
Attention Mr. Edward F. Strong
Chief of Engineering
Laboratories

Convair
Division of General Dynamics
Corporation
Fort Worth, Texas
Attention Mr. James F. Robinson
Chief of Engineering Test
Laboratories

Convair Astronautics Division
General Dynamics Corporation
San Diego 12, California
Attention Mr. James F. Watson
Senior Research Engineer

Coulter Steel and Company
Berkeley, California
Attention Mr. P. Freeman

Crucible Steel Company of America
Midland Research Laboratory
P. O. Box 226
Midland, Pennsylvania
Attention Mr. W. W. Wentz, Manager
Tech. Development
Refractory Metals

Curtiss-Wright Corporation
Metals Processing Division
Buffalo 15, New York
Attention Mr. Thomas Weidig, Manager
Metallurgical Laboratory

Curtiss-Wright Corporation
Wright Aeronautical Division
Wood Ridge, New Jersey
Attention Mr. D. I. Macfarlane

Damascus Tube Company
P. O. Box 71
Greenville, Pennsylvania
Attention Mr. Byron Burd
Chief Metallurgist

Douglas Aircraft Company, Inc.
El Segundo, California
Attention Mr. R. H. Gassner
Chief Metallurgist

Douglas Aircraft Company, Inc.
3855 Lakewood Boulevard
Long Beach, California
Attention Mr. L. O. Curtis
Materials Engineer

Douglas Aircraft Company
3000 Ocean Park Boulevard
Santa Monica, California
Attention Mr. Ed. P. Troeget, Chief
Materials Research &
Process Engineering

Douglass Aircraft Company, Inc.
Tulsa 15, Oklahoma
Attention Mr. J. E. Garol, Chief
Materials and Process
Engineering

E. I. duPont de Nemours and Company, Inc.
Curtis Bay - 2001 Benhill Avenue
Baltimore 22, Maryland
Attention Mr. J. A. Crane, Laboratory
Engineer, Metal Products

E. I. duPont de Nemours and Company, Inc.
Pigments Department
Experimental Station
Wilmington 98, Delaware
Attention Dr. B. Mahla

Extrusions, Inc.
P. O. Box 322
Caldwell, New Jersey
Attention Mr. John W. Howery
Chief Engineer

Fairchild Engine and Airplane
  Corporation
NEPA Division
Oak Ridge, Tennessee
Attention Mr. F. K. Lampson

Fansteel Metallurgical Corporation
2200 Sheldon Road
North Chicago, Illinois
Attention Dr. A. Michaels
  Assistant Director
  Metallurgical Research

Firth Sterling Incorporated
3113 Forbes Street
Pittsburgh 30, Pennsylvania
Attention Dr. C. H. Toensing

General Electric Company
Hanford Atomic Products Division
Richland, Washington
Attention Mr. Paul Kingsly
  217 T Building - 200 Area

General Electric Company
3198 Chestnut Street
Philadelphia 4, Pennsylvania
Attention Mr. Robert F. Koenig
  Manager, Materials & Processes Laboratory, Missiles & Ordnance Systems Department

General Electric Company
Central Research
Schenectady, New York
Attention Mr. D. W. Lille

General Electric Company
Applied Research Operations
Flight Propulsion Lab Department
Cincinnati 15, Ohio
Attention Mr. Louis P. Jahnke
  Manager, Metallurgical Engineering

General Electric Company
Missile and Space Division
Evendale, Ohio

General Electric Company
Aircraft Gas Turbine Division
Large Jet Engine Department
Building 501
Cincinnati 15, Ohio
Attention Mr. G. J. Wile
  Engineering Manager
  Metallurgical Engineering Operations

General Electric Company
21800 Tungsten Road
Cleveland 17, Ohio
Attention Mr. Howard Green

Goodyear Aircraft Corporation
1210 Massillon Road
Akron 15, Ohio
Attention Mr. Robert S. Ross, Manager
  Aeromechanics R & D

Grumman Aircraft Corporation
Plant 12
Bethpage, L.I., New York
Attention Mr. A. R. Mead, Manufacturing
  Staff Engineer

Harvey Aluminum, Incorporated
19200 South Western Avenue
Torrance, California
Attention Mr. G. A. Moudry
  Technical Director

Haynes Stellite Company
Kokomo, Indiana
Attention Mr. E. B. Bitzer, Jr.

Hughes Aircraft Company
Culver City, California
Attention Mr. Glenn L. Robinson
  Member of Technical Staff

Hunter Douglas Aluminum Corporation
3016 Kansas Avenue
Riverside, California
Attention Mr. Robert A. Kaser
  Engineering Superintendent
  Research Department

Jet Propulsion Laboratory
California Institute of Technology
4800 Oak Grove Drive
Pasadena 3, California
Attention Mr. I. E. Newlan

Kawecki Chemical Company
Boyertown, Pennsylvania
Attention Mr. H. C. Kawecki
President

Kennametal, Incorporated
Lloyd Avenue
Latrobe, Pennsylvania
Attention Mr. E. N. Smith
Technical Director

Ladish Company
5481 Packard Avenue
Cudahy, Wisconsin
Attention Mr. Raymond Bloom
Chief Metallurgist

Lockheed Aircraft Corporation
3251 Hanover Street
Palo Alto, California
Attention Mr. Roger Perkins
Metallurgy and Ceramics
Research

Lockheed Aircraft Corporation
Missiles and Space Division
Sunnyvale, California
Attention Mr. Joseph F. Zechmeister
Manager, Test Lab Services

Lockheed Aircraft Corporation
Missiles Systems Division
Sunnyvale, California
Attention Mr. Clayton O. Mathews

Marquardt Aircraft Company
16555 Saticay Street
Van Nuys, California
Attention Mr. Joseph W. Chambers
Supervisor, Materials
and Process Section

Martin Company
Baltimore 3, Maryland
Attention Mr. Fred D. Jewett
Structures Staff Engineer

Martin Company
Denver Division, Mail No. L-9
P. O. Box 179
Denver 1, Colorado
Attention Mr. K. R. Agricola

Massachusetts Institute of Technology
Cambridge 39, Massachusetts
Attention Prof. N. J. Grant, Metallurgy

Materials Advisory Board
National Academy of Sciences
2101 Constitution Avenue, N.W.
Washington 25, D.C.
Attention Dr. J. Lane

Materials Central
Aeronautical Systems Division
Wright-Patterson Air Force Base,
Ohio
Attention Mr. I. Perlmutter, Chief
Physical Metallurgy Branch

McDonnell Aircraft Corporation
Lambert-St. Louis Airport
P. O. Box 516
St. Louis 3, Missouri
Attention Mr. Emil G. Szabo
Manager Production
Engineering

National Aeronautics and Space Admin.
21000 Brookpark Road
Cleveland 35, Ohio
Attention Mr. G. M. Ault

National Aeronautics and Space Admin.
Plumbrook Reactor Facility
Sandusky, Ohio
Attention Mr. Charles B. Hartley

National Research Corporation
70 Memorial Drive
Cambridge 42, Massachusetts
Attention Dr. M. L. Torti

Naval Research Laboratory
Washington 25, D.C.
Attention Mr. W. S. Pellini
Superintendent, Metallurgical
Division

New York University
Collete of Engineering
New York 53, New York
Attention Dr. George Gerard
Assistant Director
Research Division

North American Aviation, Inc.
Rocketdyne Division
Canoga Park, California
Attention Mr. W. T. Chandler
Principle Scientist
Metallurgy

Naval Reactor Branch
U. S. Stomic Energy Commission
Washington 25, D.C.
Attention Mr. R. W. Staehle

North American Aviation, Inc.
Missile Division
12214 Lakewood Boulevard
Downey, California
Attention Mr. R. F. Nicholas
Supervisor of Advanced
Engineering

North American Aviation, Inc.
Los Angeles Division
International Airport
Los Angeles 45, California
Attention Mr. L. P. Spalding
Section Head, Materials

Northrop Aircraft Corporation
1001 East Broadway
Hawthorne, California
Attention Mr. Royal B. Jackman
Chief, Engineering
Laboratories

Nuclear Metals, Inc.
224 Albany Street
Cambridge 39, Massachusetts
Attention Dr. A. R. Kaufman

Oak Ridge National Laboratory
Oak Ridge, Tennessee
Attention Mr. Peter Pantriaka

Office of Naval Research
Navy Department
Washington 25, D.C.
Attention Code 423

Oregon Metallurgical Corporation
Albany, Oregon
Attention Mr. S. Shelton

Pratt and Whitney Aircraft
Connecticut Aircraft Nuclear Engine Lab.
P. O. Box 611
Middletown, Connecticut
Attention Mr. L. M. Raring, Chief
Metallurgical and
Chemical Labs

Reactive Metals, Inc.
980 Warren Avenue
Niles, Ohio
Attention Mr. O. Bertea, Process
Engineer

Republic Aviation Corporation
Farmingdale, L.I., New York
Attention Mr. E. A. Simkovich

Shieldalloy Corporation
W. Boulevard
Newfield, New Jersey
Attention Mr. John J. Collins
Technical Sales

Special Metals Corporation
New Hartford, New York
Attention Mr. F. N. Daranara

Stauffer Chemical Company
636 California Street
San Francisco 8, California
Attention Mr. C. L. Arnold,
Vice President of
Research

Stauffer Metals Company
Richmond, California
Attention Mr. J. K. Hiem

Superior Tube Company
Norristown, Pennsylvania
Attention Mr. Hugh W. Cooper, Director
Mechanical Development Div.

Temescal Metallurgical Corporation
Richmond, California
Attention Dr. C. d'A Hunt

Thompson-Ramo-Wooldridge, Inc.
23555 Euclid Avenue
Cleveland 17, Ohio
Attention Mr. A. S. Nemy
Engineering Supervisor

Union Carbide Nuclear Company
X-10 Laboratory Record Department
P. O. Box X
Oak Ridge, Tennessee
Attention Mr. W. D. Manley

United Aircraft Corporation
Pratt and Whitney Division
East Hartford 8, Connecticut
Attention Mr. Winston H. Sharp
Metallurgist
Engineering Department

United States Atomic Energy Commission
Germantown, Maryland
Attention: Mr. Socrates Christopher

Universal-Cyclops Steel Corporation
Refractomet Division
Bridgeville, Pennsylvania
Attention Mr. P. C. Rossin
General Manager

University of California
Los Alamos Scientific Laboratory
P. O. Box 1663
Los Alamos, New Mexico
Attention Dr. R. P. Hammond

University of California
Laurence Radiation Laboratory
Livermore, California
Attention Mr. Carl Schweichert

Viking Forging Company
544 Cleveland Avenue
Albany, California
Attention Mr. V. Walberg

Wah Chang Corporation
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